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# The Journal
## *of the*
# Society of Dyers and Colourists

Volume 73 Number 8

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THE SOCIETY OF DYERS AND COLOURISTS
DEAN HOUSE PICCADILLY BRADFORD YORKSHIRE

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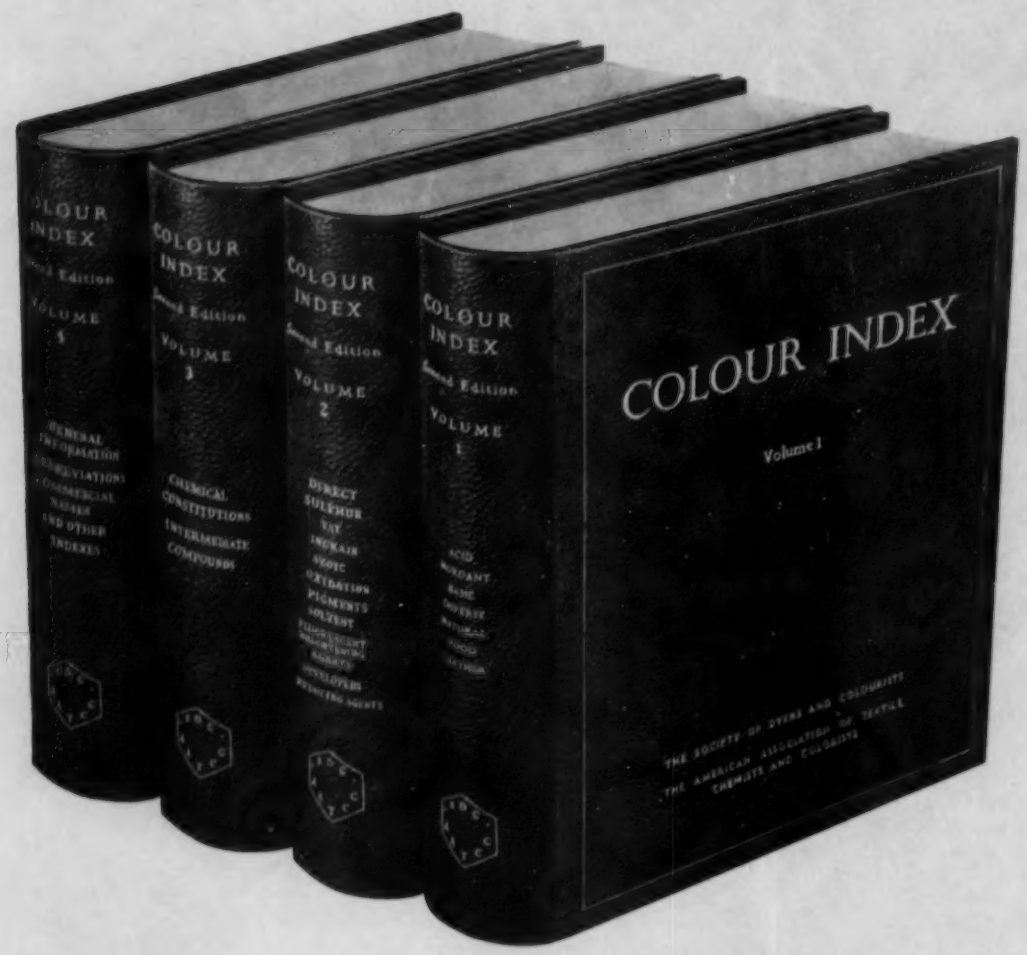


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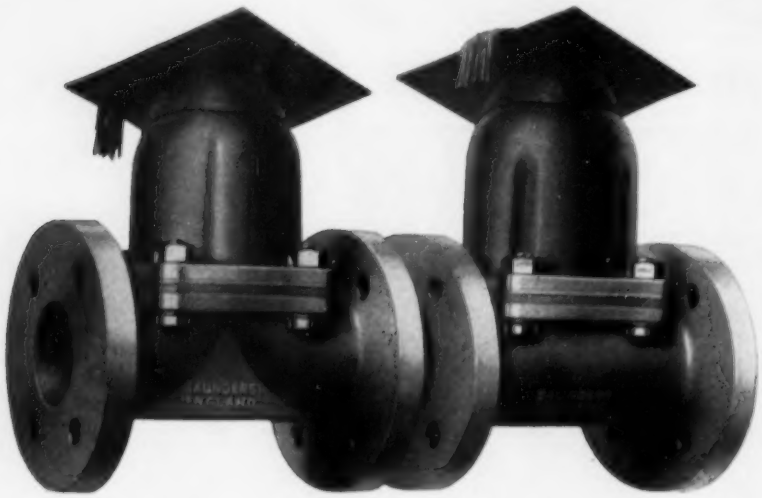

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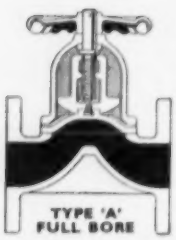


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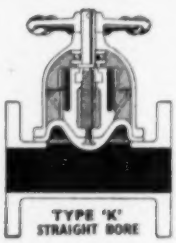


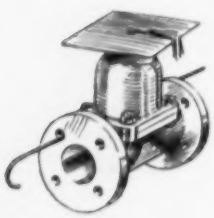

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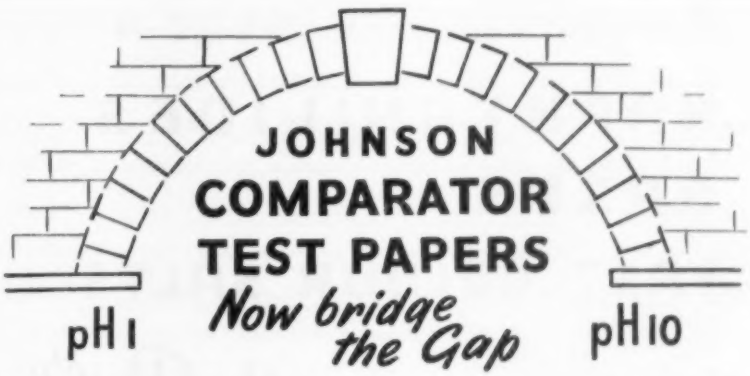


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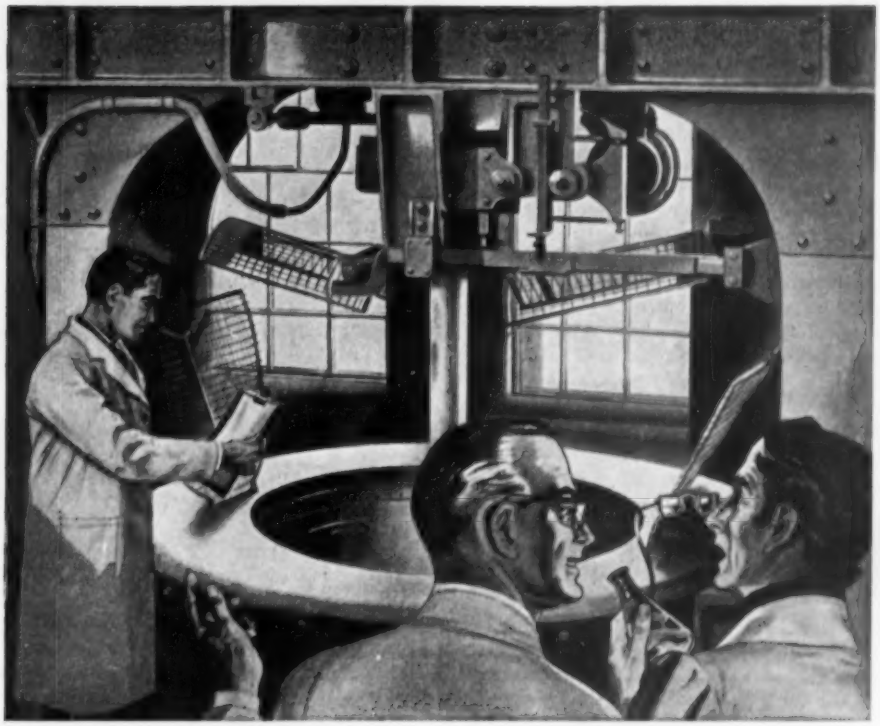

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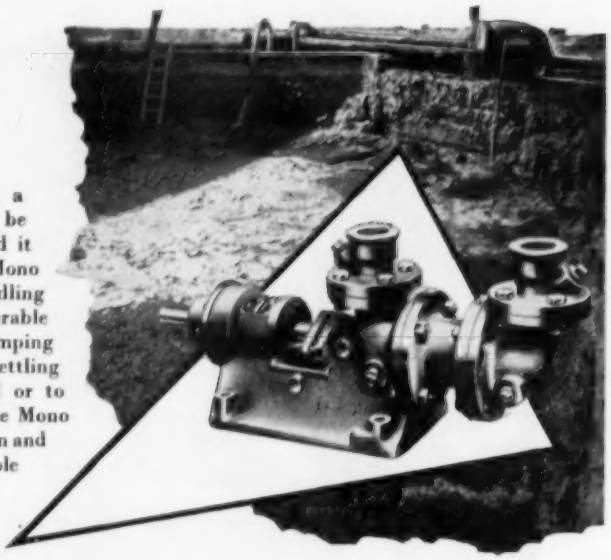

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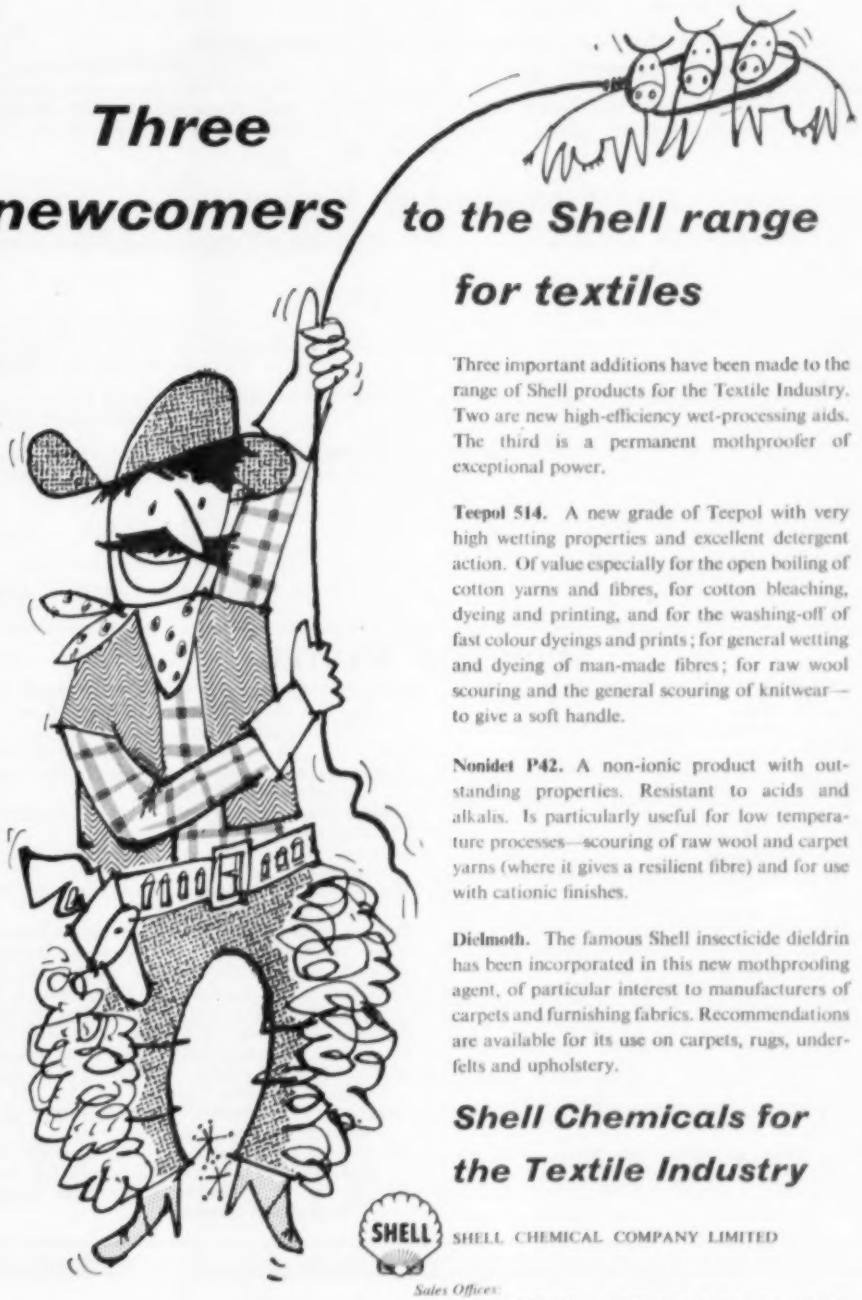

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**NOTICE TO MEMBERS AND SUBSCRIBERS**

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–7 of the January 1957 and pages 285–290 of the July 1957 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

# Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

COMMUNICATIONS

Reclamation of Fibres from Rags— I and II — *J. C. Atkinson and J. B. Speakman*

The Variation of Colour Fastness on Fabrics — *S. Horrdin and Ulf Bülow*

The Use of Paper Chromatography in the Recognition of Chemical Damage to Wool — *V. Köpke and B. Nilssen*

The Preparation and Properties of Regenerated Cellulose containing Vinyl Polymers
III— Moisture Relations — *G. Landells, J. A. Leathley, and C. S. Whewell*

The Effect on Wool of Boiling in Aqueous Solutions
III— Acids and Acidic Salt Solutions — *R. V. Peryman*

Relative Stabilities of Metal Derivatives of *oo'*-Dihydroxyazo Dyes — *F. A. Snavely, W. C. Fernelius, and B. E. Douglas*

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| WATER-REPELLENT STAIN-RESIST | Cotton<br>Viscose | BT.323 |
| CRISP OR STIFF | Cotton<br>Viscose | BT.6 or BT.318 or BT.6 plus starch or BT.322 plus starch |
| | Nylon/"Terylene" | BT.322 or BT.321 or BT.309 |
| COMBINED CREASE RESIST AND STAIN RESIST | Cotton<br>Viscose | BT.323 plus BT.322 |
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# FORTHCOMING MEETINGS OF THE SOCIETY

**Tuesday, 24th September 1957**

NORTHERN IRELAND SECTION. *The Uses of Acrilan in 100% Form as well as in Blends with Other Fibres, and The Dyeing and Finishing of Such Materials.* W. H. Hindle Esq. (Technical Director, Chemstrand Ltd., U.S.A.), Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

SCOTTISH SECTION. *Some Experiments on the Effect of Dye, Fibre and Atmosphere on Light Fastness.* Dr. G. Schmidt (Badische Anilin & Soda Fabrik). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

**Wednesday, 2nd October 1957**

MIDLANDS SECTION. *The Shrink Resistance of Wool.* A. N. Davidson, Esq., B.Sc. College of Technology, **Leicester.** 7 p.m.

**Friday, 4th October 1957**

LONDON SECTION. *Flame-proofing of Textile Fabrics, with particular reference to the function of Antimony compounds.* N. J. Read, Esq., A.C.G.I., F.R.I.C. (Associated Lead Manufacturers Ltd.). (Joint meeting with the London Branch of the Clothing Institute.) Royal Society, Burlington House, **London, W.1.** 6 p.m.

**Friday, 11th October 1957**

LONDON SECTION. Fifth London Lecture. *The Art of Costume.* Miss Anne M. Buck, Keeper of the Gallery of English Costume, Manchester. Waldorf Hotel, **London, W.C.2.** 7 p.m.

WEST RIDING SECTION. Supper and Smoker. Victoria Hotel, **Bradford.** 7.30 for 8 p.m.

**Tuesday, 15th October 1957**

SCOTTISH SECTION. *Dyeing and Finishing of Fibro Blends.* W. Lonie, Esq., B.Sc., Ph.D. (British Rayon Research Association). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

**Wednesday, 16th October 1957**

SCOTTISH JUNIOR BRANCH. *Dyeing and Finishing of Fibro Blends.* W. Lonie, Esq., B.Sc., Ph.D. (British Rayon Research Association). Technical College, **Paisley.** 7.30 p.m.

**Thursday, 17th October 1957**

MANCHESTER JUNIOR BRANCH. *The Scope of the Dyeing Technologist in the Textile Industry.* H. A. Thomas, Esq., B.Sc., Ph.D., F.R.I.C., F.T.I. Manchester College of Technology, **Manchester.** 4.30 p.m.

**Friday, 18th October 1957**

NORTHERN IRELAND SECTION. Dinner Dance. Woodbourne House Hotel.

**Saturday, 19th October 1957**

MIDLANDS SECTION. Ladies' Evening. Dinner Dance. Grand Hotel, **Leicester.**

**Wednesday, 30th October 1957**

MIDLANDS SECTION. *New Developments in the Winch Dyeing of Cotton Knitted Fabrics.* C. Newton, Esq., A.M.C.T. King's Head Hotel, **Loughborough.** 7 p.m.

**Thursday, 31st October 1957**

WEST RIDING SECTION. *The Dyeing of Acrilan and its Blends.* H. R. Hadfield, Esq., M.Sc.Tech. (Imperial Chemical Industries Ltd.). Victoria Hotel, **Bradford.** 7.30 p.m.

**Friday, 1st November 1957**

LONDON SECTION. *Problems of Early English Calico Printing.* Peter Floud, Esq., C.B.E., Keeper of Circulation, Victoria and Albert Museum. (Joint meeting with the London Section of the Textile Institute.) Royal Society, Burlington House, **London, W.1.** 6 p.m.

**Tuesday, 12th November 1957**

SCOTTISH SECTION. *New Methods for the Assessment of the Dyeing Characteristics of Neutral-dyeing Wool Dyes.* Dr. H. R. Hirsbrunner (Geigy Co. Ltd., Basle). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

This paper will contain many interesting data on the dyeing characteristics of the metal-complex dyes compared with those of the Polar type, and in addition will present a new interpretation of the exhaustion properties of these dyes under varying conditions.

**Tuesday, 12th November 1957**

NORTHERN IRELAND SECTION. *The Dyeing and Finishing of Fibro Blends* W. Lonie, Esq., B.Sc., Ph.D. (British Rayon Research Association). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

**Wednesday, 13th November 1957**

SCOTTISH JUNIOR BRANCH. *Synthetic Detergents in the Textile Industry.* H. Carter, Esq., A.R.I.C. (Shell Chemicals Ltd.). Technical College, **Paisley.** 7.30 p.m.

**Thursday, 14th November 1957**

MANCHESTER JUNIOR BRANCH. *Bleaching.* (The Bleachers Association.) Manchester College of Technology, **Manchester.** 4.30 p.m.

WEST RIDING SECTION. *New Methods for the Assessment of the Dyeing Characteristics of Neutral Dyeing Wool Dyes.* Dr. Hirsbrunner (Geigy Co. Ltd., Basle). Victoria Hotel, **Bradford.** 7.30 p.m.

**Tuesday, 19th November 1957**

MIDLANDS SECTION. *Flameproofing.* T. H. Morton, Esq., M.Sc., Ph.D., F.S.D.C. and F. Ward, Esq. Gas Board Theatre, **Nottingham.** 7 p.m.

**Tuesday, 26th November 1957**

NORTHERN IRELAND SECTION. *Terylene Polyester Fibre in the Linen Industry.* P. W. Eggleston, Esq., B.Sc., A.T.I. (Imperial Chemical Industries Ltd.). (Joint Meeting with the Textile Institute.) Kensington Hotel, **Belfast.** 7.30 p.m.

**Thursday, 28th November 1957**

WEST RIDING SECTION. *Problems in the Dyeing of Fast to Washing Colours on Wool Yarn.* J. F. Gaunt, Esq., Ph.D., F.R.I.C., F.T.I. (Patons & Baldwins Ltd.). Metropole Hotel, King Street, **Leeds.** 7.30 p.m.

**Tuesday, 3rd December 1957**

SCOTTISH JUNIOR BRANCH. *The Measurement of Damage to Wool Fibres.* J. C. Brown, Esq. (Clayton Dyestuffs Co. Ltd.). Technical College, **Paisley.** 7.30 p.m.

**Friday, 6th December 1957**

LONDON SECTION. Details later.

**Tuesday, 10th December 1957**

NORTHERN IRELAND SECTION. *An Impartial Observer's View of the Bleaching and Dyeing Industry—A Consultant Speaks Out.* T. L. Collyer, Esq., C.B.E., A.M.I.Mech.E., A.M.I.I.A. Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

SCOTTISH SECTION. *Dyeing of Acrilan.* H. R. Hadfield, Esq., M.Sc.Tech. (Imperial Chemical Industries Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Recent developments in the dyeing of Acrilan will be reviewed and the dyeing and fastness properties of acid, chrome and disperse dyes will be detailed. The dyeing of Acrilan blends will be briefly reviewed.

**Wednesday, 11th December 1957**

MIDLANDS SECTION. *The Application of Amino Resin Finishes to Cotton.* A. R. Smith, Esq., B.Sc. King's Head Hotel, **Loughborough.** 7 p.m.

**Thursday, 12th December 1957**

MANCHESTER JUNIOR BRANCH. *The Textile Finisher.* F. Wood, Esq. Manchester College of Technology, **Manchester.** 4.30 p.m.

WEST RIDING SECTION. *Watering of Acetate Poults.* E. France, Esq., B.Sc., A.R.C.S., A.M.C.T. (Courtaulds Ltd., Bocking). Victoria Hotel, **Bradford.** 7.30 p.m.

**Friday, 10th January 1958**

LONDON SECTION. *Colour for the Dyer.* Professor W. D. Wright (Imperial College of Science and Technology). (Joint meeting with the London Section of the Oil and Colour Chemists' Association.) Royal Society, Burlington House, **London, W.1.** 6 p.m.

**Tuesday, 14th January 1958**

NORTHERN IRELAND SECTION. *The Chemical Foundations of Fast Colour Printing.* A. S. Fern, Esq., B.Sc., D.I.C., F.S.D.C. (Imperial Chemical Industries Ltd.). Further details later.

**Thursday, 16th January 1958**

MANCHESTER JUNIOR BRANCH. *High Temperature Dyeing on Synthetic Fibres.* H. R. Hadfield, Esq., M.Sc.Tech. Manchester College of Technology, **Manchester.** 4.30 p.m.

*continued on page* xxxv

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# Proceedings of the Society

## The Continuous Dyeing of Secondary Cellulose Acetate, Cellulose Triacetate, and Terylene Polyester Fibre by the Vapocol Process

D. A. Garrett

*Meetings of the Midlands Section held at the King's Head Hotel, Loughborough, on 3rd October 1956, Mr. C. A. Mills in the chair; of the Manchester Section held at the Textile Institute, Manchester, on 18th January 1957, Mr. J. W. Reidy in the chair; and of the Bradford Junior Branch held at the Technical College, Bradford, on 15th October 1956, Mr. D. Young in the chair*

A continuous dyeing technique is described in which fabrics of secondary cellulose acetate, cellulose triacetate, and Terylene are padded with disperse dyes and, after being dried, are subsequently immersed in the saturated vapour of trichloroethylene for times up to 60 sec. to fix the dyes on the fibres. The exhaustion and the fastness properties of dyes fixed by this means on secondary cellulose acetate and cellulose triacetate fibres are at least equal to those obtained by conventional dyeing methods; dyeings on Terylene are limited to pale and medium depths on filament fabrics. The construction and the operation of a continuous plant handling 12-in. wide fabrics at speeds up to 10 yd./min. are described. The process is suitable, without modification, for dyeing blends of these fibres with viscose rayon and cotton, making use of reactive dyes for the cellulosic component.

### INTRODUCTION

In the search for ways of decreasing dyeing costs, greater attention has been directed in recent years to consideration of costs other than those of the dyes themselves, such as costs of equipment, of labour, and of services. This has resulted in increased interest in continuous dyeing processes, by means of which the output per machine can be increased many times. All such systems have certain features in common, and it is useful to consider what features are essential to any continuous dyeing system in order that it may be commercially successful.

In the first place the system must operate at a speed sufficiently greater than that of a conventional batchwise process to justify the capital outlay on new equipment. This generally means operating at 30–50 yd./min., giving a five- or ten-fold advantage over, say, a single jigger or winch. In order to achieve this rate of production in a machine of reasonable physical dimensions, it is clearly essential to reduce the time of dyeing to a small fraction of that common on conventional systems; at the same time the quality of the resulting dyeings must not be in any way impaired. This can be done in two ways— by speeding up the transfer of dye to the fibre surface and by increasing the rate of diffusion of the dye within the fibre.

The first of these objectives can be achieved by increasing the concentration of dye in solution near the fibre surface, in other words, by decreasing the liquor ratio and possibly also by increasing the solubility of the dye in whatever liquid dyeing medium may be used. The second objective is achieved by raising the temperature of the fibres well above 100°c. or by using a swelling agent. The pad–steam process for application of soluble dyes to cotton or viscose rayon meets both requirements, by limiting the liquor present on the fabric so that the liquor ratio is close to unity, by providing a dyeing medium in which the dyes are readily soluble, and by bringing the fibres into contact with steam, which is a very effective swelling agent for cellulose. Hydrophobic fibres such as secondary cellulose acetate, cellulose triacetate, and Terylene, however, are not swollen appreciably by steam at about 100°c., and the disperse dyes used to colour these fibres are practically insoluble in water, so that the pad–steam process is not very effective with these fibres.

Clearly, what is required is an alternative dyeing medium which will both swell these hydrophobic

fibres and dissolve disperse dyes appreciably. Such solvents are trichloroethylene and the related chlorinated aliphatic hydrocarbons, and reference to their use for this purpose has been made by Mellor and Olpin [1, 2]. These solvents not only fulfil the requirements for a continuous dyeing process laid down earlier but also are unique in the manner in which they may be controlled in vapour form. The vapours have a density 4–5 times that of air, and may be controlled in an unsealed vessel by means of a cooling coil arranged around the inside rim of the vessel. This is the principle employed in metal-degreasing plants, in which trichloroethylene liquid is boiled in the bottom of a galvanised iron tank, the vapours displacing the air from the tank and filling it completely up to the level of a set of copper condensing coils. The leakage of solvent vapour from such a tank is low and the tank may be safely operated in workshops and similar premises, provided that adequate ventilation is maintained. It is evident that solvent vapours which may be controlled in this fashion offer an advantage over other solvents requiring totally enclosed vessels, into which any fabric to be treated must be fed through complicated seals.

The physical properties of trichloroethylene which are of particular interest in this connection are given in Table I.

TABLE I

**Physical Properties of Trichloroethylene**

| | |
|---|---|
| Boiling point ... ... ... ... | 86·7°c. |
| Specific heat ... ... ... ... | 0·223 cal./g. |
| Latent heat of vaporisation ... ... | 57·3 cal./g. |
| Density ... ... ... ... ... | 1·47 g./ml. |
| Boiling point of azeotropic mixture with water ... ... ... ... | 73·2°c. |
| Composition by weight of azeotropic mixture, trichloroethylene : water ... | 13·4 : 1 |

Apart from providing adequate ventilation in the vicinity of the vapour tank, certain other precautions should be taken to ensure that no hazards or solvent wastage arise. These precautions are elaborated elsewhere [3], but the principal ones to observe may be stated as follows—

- Unnecessary inhalation of the vapour should be avoided
- Operatives should not lean into any vessel containing solvent (liquid and vapour)
- Solvent vapour should not be allowed to come into contact with flames or red-hot surfaces, as phosgene and other acidic gases may be produced
- Contact with the skin should be avoided.

The sequence of operations in the Vapocol process, in which trichloroethylene vapour is used as the dyeing medium, is to pad the fabric with an aqueous dispersion of the disperse dye, dry the fabric, and subsequently immerse it in solvent vapour in a tank such as that described above. A simple calculation shows that, if dry Terylene fabric at room temperature is immersed in saturated trichloroethylene vapour, the weight of solvent which condenses in heating the fabric to the temperature of the vapour (87°c.) is approx. 35% of the weight of the fabric. Previous (unpublished) work by the author on the use of trichloroethylene in emulsion form as a carrier for Terylene in aqueous dyebaths has shown that the amount of trichloroethylene absorbed by the fibres is approx. 15%. This means that in the vapour tank roughly 20% of trichloroethylene condenses on the fabric in excess of that absorbed in the fibres. This excess solvent dissolves the disperse dye padded on the fabric, and the dye then diffuses rapidly into the solvent-swollen fibres. Similar amounts of trichloroethylene condense on, and are absorbed by, secondary cellulose acetate and cellulose triacetate fibres. It is of interest to note here that, because the trichloroethylene wets the fibres very readily and also dissolves the dye, the condensed solvent has a marked levelling action, tending to correct small padding faults.

The reason why it is necessary to dry the fabric prior to immersion in the solvent vapour is that the thermal capacity of wet fabric is such that more trichloroethylene condenses on the fabric in heating it to the temperature of the vapour than the fabric structure can contain, with the result that excess solvent drips from the fabric, carrying with it the dye.

After immersion of the padded fabric in trichloroethylene vapour, during which time the dye is fixed on the fibres, it is necessary to remove the solvent completely from the fabric. This can be done by raising the temperature of the fabric to the boiling point of the solvent either by means of hot air or by immersion of the fabric in water at 85–95°c. for 15–30 sec. Very rapid and complete removal of the solvent is effected by these means, and in addition a slight increase in exhaustion often occurs at this stage.

The effectiveness of this dyeing method will now be considered for the various individual fibres to which it is applicable.

### SECONDARY CELLULOSE ACETATE

The exhaustion and the build-up of disperse dyes on secondary cellulose acetate in trichloroethylene vapour were investigated by suspending small padded and dried samples of satin fabric in a vapour tank for varying times and subsequently plunging them into water at 85°c. to remove the trichloroethylene. The fabrics were then soaped at 40°c. for 15 min. in Lissapol C (2 g./litre) in order to remove unfixed dye. The vapour tank used for these experiments consisted simply of a large glass beaker in which a small volume of trichloroethylene was boiled continuously. The vapours were condensed by means of a four-turn copper cooling coil, mounted just below the inner rim of the beaker, through which cold water was circulated.

Tests were carried out to determine the effect of thickening agents, and any other additions to the pad trough liquor, on the exhaustion of dye in the vapour tank. Estimations of the dye on the fabric before and after the dyeing treatment were made by dissolving the fibre in acetone and estimating the dye concentration by measurements of optical density with the Spekker (Hilger) absorptiometer.

TABLE III

| Dye Name | *Colour Index* No. | Padding Concn. (g./kg.) | Secondary Cellulose Acetate: Dyeing Time (sec.) | Secondary Cellulose Acetate: Pick-up on Padding (% on wt. of fabric) | Secondary Cellulose Acetate: Dye fixed by Vapour Immersion (% on wt. of fabric) | Secondary Cellulose Acetate: Exhaustion (%) | Cellulose Triacetate: Dyeing Time (sec.) | Cellulose Triacetate: Pick-up on Padding (% on wt. of fabric) | Cellulose Triacetate: Dye fixed by Vapour Immersion (% on wt. of fabric) | Cellulose Triacetate: Exhaustion (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| Duranol Brilliant Yellow 6G 300 Powder Fine | C.I. Disperse Yellow 13 | 30 | — | — | — | — | — | — | — | — |
| | | 90 | 20 | 4·63 | 2·88 | 62·2 | — | — | — | — |
| Dispersol Fast Yellow A 200 Paste | C.I. Disperse Yellow 1 | 50 | — | — | — | — | 30 | 2·85 | 2·64 | 92·3 |
| | | 150 | 15 | 8·43 | 7·66 | 91·0 | | 5·55 | 5·33 | 96·0 |
| Dispersol Printing Yellow GS Paste | C.I. Disperse Yellow 3 | 50 | — | — | — | — | 20 | 2·87 | 2·74 | 95·3 |
| | | 150 | 15 | 8·7 | 7·55 | 86·7 | | 4·59 | 4·32 | 94·2 |
| Dispersol Fast Yellow GR 300 Powder Fine | C.I. Disperse Yellow 39 | 30 | — | — | — | — | 60 | — | — | — |
| | | 90 | 60 | 4·83 | 3·17 | 65·7 | | 4·82 | 3·65 | 76·0 |
| Dispersol Fast Orange AS Paste | C.I. Disperse Orange 1 | 60 | — | — | — | — | 120 | 3·69 | 3·45 | 93·6 |
| | | 180 | 30 | 12·6 | 10·4 | 82·5 | | 9·72 | 9·38 | 96·5 |
| Dispersol Fast Orange B Special Paste (150)* | C.I. Disperse Orange 13 | 60 | — | — | — | — | 30 | 4·59 | 4·53 | 98·7 |
| | | 180 | 30 | 10·6 | 8·22 | 77·5 | | 12·33 | 10·75 | 87·2 |
| Dispersol Fast Orange G 300 Powder Fine | C.I. Disperse Orange 3 | 30 | — | — | — | — | — | — | — | — |
| | | 90 | 15 | 4·97 | 3·83 | 77·2 | | — | — | — |
| Dispersol Fast Scarlet BS Paste Fine | C.I. Disperse Red 1 | 50 | — | — | — | — | 15 | — | — | — |
| | | 150 | 30 | 9·13 | 7·75 | 85·0 | | 9·54 | 9·47 | 99·3 |
| Dispersol Fast Red RS Paste Fine | C.I. Disperse Red 19 | 60 | — | — | — | — | 60 | 2·57 | 2·34 | 91·0 |
| | | 180 | 60 | 9·05 | 7·36 | 81·3 | | 7·85 | 6·4 | 81·5 |
| Duranol Red 2BS Paste Fine | C.I. Disperse Red 15 | 100 | — | — | — | — | 30 | 4·29 | 4·25 | 99·0 |
| | | 300 | 15 | 10·08 | 9·76 | 96·8 | | 8·19 | 7·58 | 92·5 |
| Duranol Red X3B 200 Paste Fine | C.I. Disperse Red 11 | 50 | — | — | — | — | 120 | 1·89 | 1·79 | 94·5 |
| | | 150 | 30 | 8·09 | 6·48 | 80·1 | | 6·06 | 5·6 | 92·3 |
| Dispersol Fast Crimson BS Paste Fine | C.I. Disperse Red 13 | 30 | — | — | — | — | 15 | — | — | — |
| | | 90 | 15 | 5·04 | 4·06 | 80·6 | | 4·74 | 4·72 | 99·5 |
| Duranol Violet RN 200 Paste Fine | C.I. Disperse Violet 14 | 50 | — | — | — | — | 60 | 2·64 | 2·22 | 84·1 |
| | | 150 | 30 | 10·35 | 8·43 | 81·5 | | 7·75 | 6·15 | 79·4 |
| Duranol Violet 2RS Paste Fine | C.I. Disperse Violet 1 | 50 | — | — | — | — | 30 | 2·68 | 2·36 | 88·2 |
| | | 150 | 30 | 8·01 | 6·99 | 87·2 | | 6·81 | 6·35 | 93·4 |
| Duranol Brilliant Violet B 300 Paste | C.I. Disperse Violet 4 | 30 | — | — | — | — | 60 | 1·8 | 1·54 | 85·3 |
| | | 90 | 60 | 6·8 | 6·07 | 89·3 | | 4·71 | 3·99 | 84·8 |
| Duranol Brilliant Violet BR 300 Powder Fine | C.I. Disperse Violet 8 | 30 | — | — | — | — | 60 | 1·61 | 1·31 | 81·5 |
| | | 90 | 60 | 6·08 | 4·42 | 72·5 | | 5·92 | 2·47 | 41·7 |
| Duranol Brilliant Blue B 200 Paste Fine | C.I. Disperse Blue 3 | 50 | — | — | — | — | 30 | 2·3 | 2·15 | 93·4 |
| | | 150 | 60 | 9·5 | 8·25 | 86·8 | | 6·99 | 6·76 | 96·8 |
| Duranol Brilliant Blue CB 200 Paste Fine | C.I. Disperse Blue 1 | 150 | — | — | — | — | 60 | 5·15 | 2·27 | 44·0 |
| | | 300 | 60 | 20·2 | 8·65 | 42·8 | | 12·0 | 3·6 | 30·0 |
| Duranol Brilliant Blue G 200 Paste Fine | C.I. Disperse Blue 14 | 100 | — | — | — | — | 30 | 1·96 | 1·59 | 81·0 |
| | | 300 | 30 | 4·8 | 3·19 | 66·4 | | 3·71 | 3·26 | 88·0 |
| Duranol Printing Blue G 200 Paste | C.I. Disperse Blue 26 | 50 | — | — | — | — | 30 | 1·92 | 1·73 | 90·1 |
| | | 150 | 60 | 7·5 | 5·2 | 69·3 | | 6·49 | 5·04 | 77·6 |
| Duranol Blue 2GS Paste Fine | C.I. Disperse Blue 24 | 100 | — | — | — | — | 120 | 3·99 | 3·83 | 96·0 |
| | | 300 | 60 | 16·5 | 8·75 | 53·2 | | 13·98 | 11·4 | 81·5 |
| Duranol Printing Blue–Green BS Paste | C.I. Disperse Blue 7 | 180 | — | — | — | — | 60 | 7·59 | 2·66 | 35·0 |
| | | 300 | 60 | 17·31 | 9·0 | 52·0 | | 13·8 | 3·37 | 24·4 |

* Produced in the laboratory

It was found that the nature of the thickener had a negligible effect on the exhaustion, as is indicated in Table II.

TABLE II

**Effect of Thickener on Exhaustion**

(Fabric padded at 50°C, with Duranol Red X3B 200 Paste Fine (50 g./litre) + Perminal PP (5 g./litre) )

| Thickener | Concn. (g./litre) | Fixation of Dye (%) |
|---|---|---|
| Gum tragacanth (8%) | 50 | 97·5 |
| Nafka crystal gum (24%) | 100 | 95·0 |
| Calasec MAH | 10 | 98·4 |

It was found, however, that the selection of the thickener was important if unfixed dye was to be readily removed in the soaping bath after dyeing. Thus it was found that with fabrics padded without thickener, or with Calasec MAH as thickener, complete removal of unfixed dye could not be achieved in all cases by a simple soaping treatment, with the result that the dyed fabrics were dulled by the residual surface deposition of certain dyes. By using a thickener of high solids content such as Nafka gum, however, the unfixed dye could be completely removed in a short soaping time, presumably because of swelling of the thickener in the soaping bath. In order to ensure complete removal of unfixed dye prior to the spectroscopic estimations in instances where thickeners other than Nafka gum were used, the fabrics were given a hand scrub in the soaping bath.

The exhaustions of a range of dyes have been measured by this technique, using in each case a time of immersion in the vapour adequate to achieve equilibrium exhaustion as judged by visual assessment of fabrics dyed for different times. The results of these measurements, together with the times required to reach equilibrium, are shown in Table III.

It should be noted that the times to reach equilibrium which are quoted are not necessarily the times of dyeing which would be required to achieve good colour build-up, since the initial rate of fixation of dye in the solvent vapour was very rapid, so that an exhaustion close to equilibrium was often reached in 5 sec., after which there was a much slower approach to the final equilibrium. The significance of this will become apparent when consideration is given to the operation of a continuous dyeing process.

It will be noted that exhaustion of dyes is very good in practically all cases, the only instances of comparatively low exhaustion being dyes for which it is known that the exhaustion in aqueous dyebaths also is low. Penetration of the fibres is complete in the dyeing times indicated, and in the majority of cases is complete in 5 sec.

In order to compare the effectiveness of the Vapocol dyeing process with that of conventional dyeing methods, estimations have been made of the amounts of a range of disperse dyes padded on cellulose acetate satin fabric. The equivalent amounts of powder-brand dyes have then been applied to fabrics from aqueous dyebaths, so that a visual comparison could be made between the two methods. The results are shown in Fig. 1, from which it will be observed that the solvent vapour method of fixation is in no instance inferior to the aqueous method.

The fastness properties of a range of dyes applied by the Vapocol method have been compared with the fastnesses of aqueous-dyed fabrics, and in all instances the results are comparable, the maximum differences observed being 0·5 grade, probably due to slight differences in depth.

The mechanical properties of yarns taken from typical unset filament fabrics treated in trichloroethylene vapour have been compared with those of the untreated yarns, with the results shown in Table IV.

TABLE IV

| Fibre | Time of Treatment in Trichloroethylene Vapour (min.) | Shrinkage (%) Warp | Shrinkage (%) Weft | Weft Denier | Weft Tenacity (g./denier) | Weft Extensibility (%) |
|---|---|---|---|---|---|---|
| Secondary cellulose acetate | 0 | — | — | 136 | 1·16 | 26·7 |
| | 0·5 | 3·7 | 3·0 | 139 | 1·09 | 31·2 |
| Cellulose triacetate | 0 | — | — | 154 | 0·84 | 24·5 |
| | 0·5 | 8·3 | 4·2 | 155 | 0·85 | 25·7 |
| Terylene | 0 | — | — | 79·9 | 3·4 | 16·3 |
| | 2·0 | 0·6 | 0·7 | 81·4 | 3·3 | 16·2 |

There is no evidence in these results of the mechanical properties of the fibres being significantly impaired in any way, and the shrinkage which takes place in the solvent vapour under relaxed conditions is similar to that which occurs in normal aqueous dyebaths at 85°C.

### CELLULOSE TRIACETATE

An investigation similar to that on secondary cellulose acetate has been carried out on unset cellulose triacetate filament fabric. Again no disperse dye has been found which cannot be successfully applied by the Vapocol method, and the dyeing times and exhaustions of a range of dyes, estimated in the same manner as for the secondary acetate, have been determined and are shown in Table III.

Comparison by visual assessment of solvent-vapour-dyed and conventionally dyed fabrics reveals that on the triacetate the performance of several dyes is superior when applied by the Vapocol method (Fig. 2). It will be noted that the tinctorial yields of several dyes applied by the Vapocol method are appreciably greater than those obtained by conventional application from aqueous dyebaths at the boil. Again the fastness properties are similar by the two dyeing methods.

Mechanical tests carried out on the fabrics after immersion in the trichloroethylene vapour show

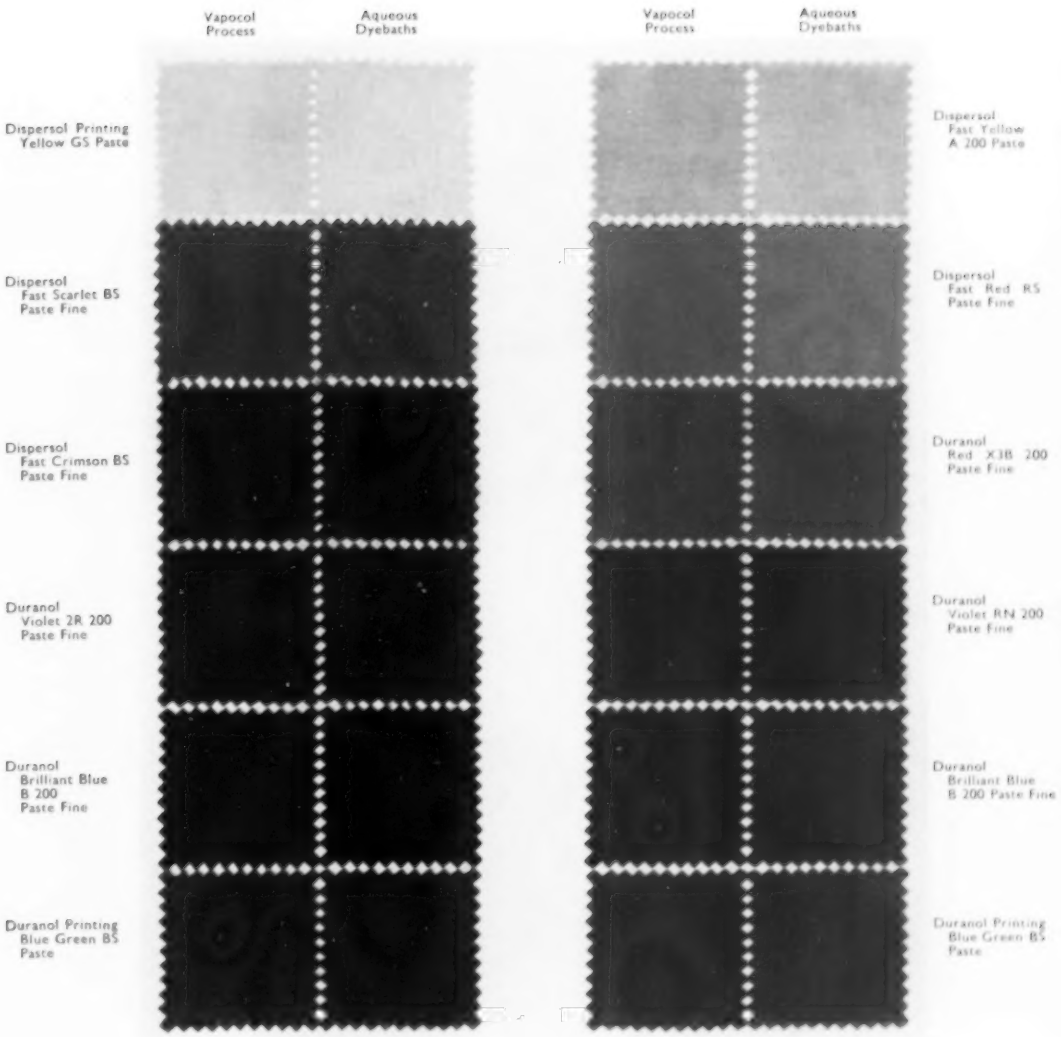
Vapocol Process
Aqueous Dyebaths
Dispersol Printing Yellow GS Paste
Dispersol Fast Scarlet BS Paste Fine
Dispersol Fast Crimson BS Paste Fine
Duranol Violet 2R 200 Paste Fine
Duranol Brilliant Blue B 200 Paste Fine
Duranol Printing Blue Green BS Paste
Vapocol Process
Aqueous Dyebaths
Dispersol Fast Yellow A 200 Paste
Dispersol Fast Red RS Paste Fine
Duranol Red X3B 200 Paste Fine
Duranol Violet RN 200 Paste Fine
Duranol Brilliant Blue B 200 Paste Fine
Duranol Printing Blue Green BS Paste

FIG. 1—Secondary Cellulose Acetate

FIG. 2—Cellulose Triacetate

FIG. 1 and 2—Comparison of Dyeings by the Vapocol Process and by Conventional Methods

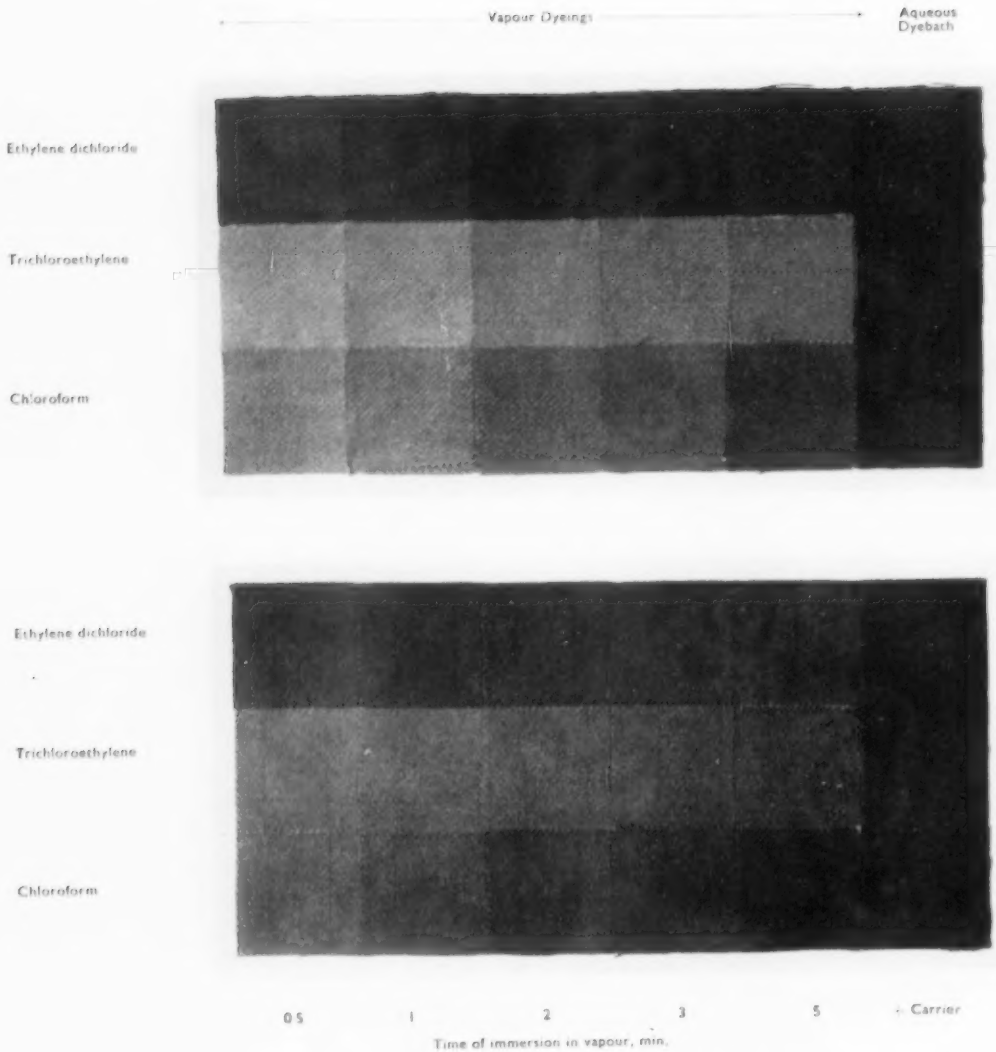


FIG. 4 and 5 — Dyeings of Durano Red N3B 200 Paste Fine and of Duranol Printing Blue G 200 Paste on Terylene Staple-fibre Fabric

no evidence of significant permanent damage to the fibres during processing (Table IV).

### TERYLENE POLYESTER FIBRE

A limited number of disperse dyes have been applied to Terylene staple-fibre fabric by the Vapocol technique*, and the rates of dyeing measured by extraction of the undyed padded material and of the padded material exposed to solvent vapour for varying times. The rate-of-dyeing curves of four dyes are shown in Fig. 3. It will be noted at once that the rate of dyeing on Terylene is very much less than on secondary cellulose acetate and cellulose triacetate, and that in order to approach equilibrium a time of immersion in the vapour of approx. 5 min. is necessary with these slow-dyeing, fast-to-sublimation dyes [4]. The exhaustions achieved at equilibrium are lower, and furthermore the measured exhaustions are not a true indication of the efficiency of the process as judged visually. Thus dyeings carried out by the Vapocol method are both weaker and duller than those obtained on fabrics dyed in aqueous dyebaths at the boil with Tumescal D as carrier, even if the amount of dye fixed on the fibres is the same in both cases. This is due partly to poor fibre penetration and partly to the fact that the fibre hairs projecting from the surface of the fabric are stripped of dye in the solvent vapour. This stripping is due to solvent condensing on the hairs, dissolving the dye, and carrying it into the bulk of the fabric. In the case of cellulose acetate and triacetate materials this does not happen, because fixation of dye is so rapid that there is no opportunity for the solvent to wash the dye from the fibres.

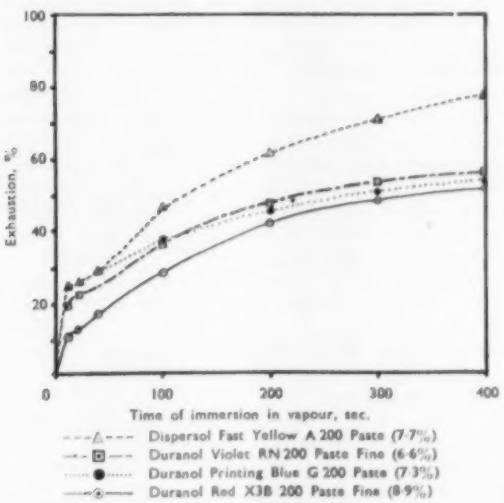


FIG. 3—Rate of Dyeing of Disperse Dyes applied by the Vapocol Process to Terylene Staple Fabric

The results achieved with the rapid-dyeing dyes such as Dispersol Fast Yellow G, Dispersol Fast Orange G, Duranol Red 2B, and Duranol Blue GN more closely approach those obtained by normal dyeing methods, but even these cannot be built up to dark colours in trichloroethylene vapour. It appears, then, that the range of colours on Terylene possible by this process is limited to pale to medium depths, and in the case of spun material attention must be paid to the effect of the stripping of surface fibres by the solvent. The tensile properties of the Terylene are unimpaired by the solvent vapour treatment.

The poor penetration of Terylene fibres indicates that the disappointing results of dyeings carried out by the Vapocol process are due to the inability of trichloroethylene to swell the fibres adequately. For this reason an examination has been made of alternative solvents, and it has been found that ethylene dichloride and chloroform vapours in particular are superior to trichloroethylene for the fixation of disperse dyes on Terylene fibres. In Fig. 4 and 5 are shown comparisons of the results achieved on exposure of Terylene staple-fibre fabrics, padded with Duranol Red X3B and Duranol Printing Blue G respectively and dried, to the saturated vapours of trichloroethylene, ethylene dichloride, and chloroform for various times. In each case the solvent has been completely removed after dyeing, and unfixed dye soaped off the fabrics. It is evident that ethylene dichloride is far superior to trichloroethylene so far as exhaustion and build-up of colour are concerned, and that the depths achieved in ethylene dichloride closely approach those of conventionally dyed fabrics. Unfortunately, whilst ethylene dichloride vapour can be controlled in the same fashion as that employed for trichloroethylene, it has a low flash-point (13°C.) and in addition can induce chronic toxicity if the solvent vapour is inhaled over long periods even at low concentrations. The maximum allowable concentration is generally accepted as 50 p.p.m. Since the minimum level of detection by smell is 60 p.p.m., this is clearly a hazardous solvent with which to work, and its use on a commercial scale cannot be contemplated until more work has been carried out on the problems of safe handling.

Alternative solvents appear to offer no advantage with cellulose acetate or triacetate materials: presumably the swelling action of trichloroethylene is sufficiently high to promote rapid diffusion of dye without excessive swelling of the fibres such as occurs with ethylene dichloride, which dissolves secondary cellulose acetate on prolonged exposure to the vapour.

### OTHER FIBRES

The application of the Vapocol technique is limited to secondary cellulose acetate, cellulose triacetate, and Terylene fibres, and no other materials so far examined can be dyed by this method, presumably owing to the inability of trichloroethylene or similar solvents to swell the fibres.

### OPERATION OF CONTINUOUS PLANT

The results so far quoted clearly indicate the applicability of the Vapocol method to a continuous dyeing process. Thus, in the case of acetate and

* Patent protection pending

triacetate fibres, the majority of colours can be achieved in times of dyeing of the order of 30 sec., since it is not always essential to reach complete equilibrium, and in all cases the colour yield is equal to or better than that achieved in a dyeing time of 1 hr. in a normal aqueous dyebath. The essential features of a continuous Vapocol dyeing plant are shown in Fig. 6, which is a simplified drawing of a small-scale plant designed to dye material of width 12 in. at speeds up to 10 yd./min.

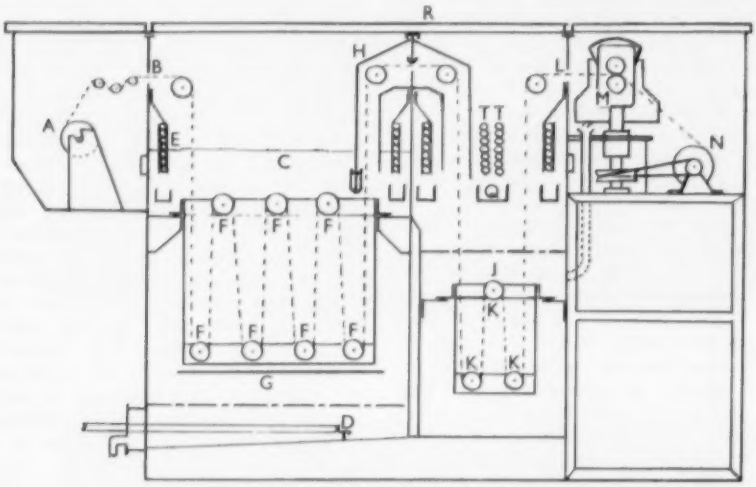


FIG. 6—Plant for Continuous Dyeing

On a full-scale plant the vapour treatment would be preceded by the padding and drying stages, all working on a continuous basis, but on the pilot-scale plant to be described the fabric has to be batched up following the padding and drying stages. From the batching roller *A* the fabric passes over tensioning bars and into the galvanised iron vapour tank *C* through the narrow slot *B*. Trichloroethylene is boiled in the bottom of *C* by means of the closed steam coil *D*, and the vapour fills the tank to the level of the copper condensing coil *E*. The fabric is supported in the vapour by the rollers *F*, so that the total length of fabric immersed in the vapour is approx. 5 yd. The rollers *F* are of mild steel covered with a protective coat of Lithcote P lacquer. A protective plate *G* is placed below the fabric in order to prevent splashing from the boiling liquid. The fabric leaving the vapour tank *C* is saturated with solvent, and in order to prevent contamination of the atmosphere in the tank above the level of the cooling coils the fabric is carried inside the duct *H* into the enclosed hot-water tank *J*, in which a length of approx. 2 yd. of fabric is immersed in the water over the rollers *K*. The fabric, freed from solvent, passes out of *J* through the slot *L* and on to the power-driven mangle *M*, which is used to draw the fabric through the equipment. It is finally batched up on the roller *N*.

The water flash-off system of solvent removal and recovery is preferred because experience has shown this to offer more efficient recovery and greater safety, since there is no possibility of solvent leakage, such as might occur from the entry and exit of, say, an air dryer. It also offers the advantage of uniform treatment of the fabric with less likelihood of listing.

The solvent vapour evaporated in the hot-water tank, together with steam, is condensed on the condensing coils *T*, and the liquid collected in the trough *Q*, whence it is carried out to a water separator. The recovered trichloroethylene is then returned to the solvent vapour tank.

Because of the degreasing action of trichloroethylene vapour, conventional lubricated bearings are unsuitable for the rollers carrying the fabric in the vapour and hot-water tanks. The rollers therefore have stainless-steel shafts running in Ferrobestos bearings. No intermediate drive has been used between the entry of the fabric into the plant and the mangle used to draw the fabric through the plant and to express excess liquid. The permissible tensions on the fabric in the vapour are discussed later (p. 371), but, on the equipment described, it has been possible to process cellulose acetate and triacetate satin fabrics with an overall extension of 1·5%, although a light-weight secondary acetate Taiho fabric exhibited an extension of 8%. Clearly, on larger-scale equipment, in which a greater number of rollers might be included, intermediate drives would be necessary to limit the build-up of warp tension in the fabric.

The machine is equipped with a loose-fitting cover *R*, which is intended to reduce still further the escape of solvent into the atmosphere and also to limit the effect of draughts on the level of the solvent in the first tank.

Secondary cellulose acetate and cellulose triacetate fabrics have been successfully dyed in the equipment described, a full range of colours being developed in times of dyeing of the order of 30 sec., which are equivalent to a speed of processing of 10 yd./min. Pale and medium depths have also been dyed on Terylene, in dyeing times of 3–5 min. No evidence has been found of listing, ending, or

marking-off on the rollers, and the quality of the resulting dyeings has been fully equal to that of conventionally dyed materials. The absence of listing or ending is to be expected because of the perfectly uniform treatment to which the fabric is subjected in the vapour tank. Thus the vapour at all points in the tank is saturated and at the boiling point of the trichloroethylene. A consequence of this is that a direct comparison is possible between fabrics treated in a continuous plant and in small-scale laboratory equipment. The physical conditions are identical in each case, and so accurate colour matching can be carried out on laboratory-scale equipment.

The temperature of the hot-water flash-off tank is important in that the higher the temperature the more rapid and complete is the removal of solvent from the dyed fabric. In the case of triacetate and Terylene a temperature of 90°c. can be employed with advantage, but in the case of secondary acetate the temperature must be limited to about 85°c. in order to avoid hydrolysis of the acetate. The lower temperature is satisfactory with this material, since the fibre structure from which the solvent has to be removed is less compact and the solvent is therefore much more readily removed. The total loss of solvent has been estimated to be less than 3% of the cloth weight processed. In the vapour tank the constant distillation of solvent confers a self-cleansing action, and slight contamination of the liquid trichloroethylene in no way affects the dyeing operation. The amount of dye lost in the solvent vapour is very small.

In the water flash-off bath some dye is removed from the fabric, and a constant flow of water into the tank may be needed on long runs, or with heavy dyeings, to limit the concentration of dye in the bath to below the level at which the colour is affected.

Fabrics dyed on this equipment have been subsequently soaped in open width on a jigger, but on the bulk scale a continuous soaping, rinsing, and drying range could be envisaged following the water flash-off treatment.

Since the fabrics are handled in open width, no setting treatment is necessary prior to dyeing, the fabrics being set after dyeing under the same conditions as those employed following conventional dyeing treatments.

### TENSILE PROPERTIES OF YARNS IN SOLVENT VAPOUR

A study has been carried out of the tensile properties of secondary acetate, triacetate, and Terylene fabrics in trichloroethylene vapour, in order to establish the maximum fabric tensions which can be tolerated in the solvent vapour tank.

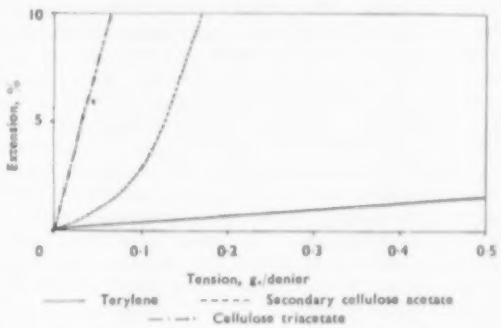


FIG. 7—Stress-Strain Curves of Filament Fibres in Trichloroethylene Vapour

Stress–strain curves (Fig. 7) measured under conditions of constant rate of loading have been obtained on continuous-filament yarn immersed in the vapour. Little or no recovery of extension results on unloading of the fabric following extension. From the initial slopes of these curves the extensions per gram load per denier have been estimated, together with the calculated extensions of fabrics having different end-counts and warp-thread deniers subjected to different tensions (Table V).

TABLE V

| Fibre (filament yarn) | Extension per g./denier (in vapour) (%) | Extension per oz. warp tension per in. width of fabric (calc.) (%) | |
|---|---|---|---|
| End count | | 300 | 100 |
| Warp yarn denier | | 75 | 100 |
| Secondary cellulose acetate | 34·0 | 0·044 | 0·097 |
| Cellulose triacetate | 153·0 | 0·18 | 0·41 |
| Terylene | 2·3 | 0·0029 | 0·0065 |

Table V may be used as a guide to the number of intermediate drives needed to limit the build-up of tension in the fabric passing through the vapour.

### DYEING OF FIBRE BLENDS

All the results so far described have been obtained with materials containing only one kind of fibre. Many fabrics, however, consist of blends of two or more fibres, and consideration has been given to the continuous dyeing of these materials. Since the technique is intended to be operated as a continuous process, interest is limited to those blends occurring in large quantities. Attention has therefore been principally directed to blends with cellulosic fibres, such as viscose rayon and cotton, in which the cellulosic component is generally dyed with direct dyes.

In order to fix direct dyes continuously the pad-steam process can be used, the direct and disperse dyes being padded together and the fabrics subjected to a steaming treatment directly following the vapour tank. By this means a range of solid shades and cross-dyed effects have been obtained.

The disadvantage of this process is that an additional steaming stage is required, and since the solvent is removed in the steaming chamber a problem arises from contamination of the steam by the trichloroethylene. A much more satisfactory approach has therefore been adopted making use of the Procion dyes to dye the viscose rayon or cotton components. The sequence of operations is to pad the disperse dyes together with the Procion dyes and sodium bicarbonate, and then dry the fabric at about 105°C. At this temperature, fixation of the Procion dyes occurs, and the disperse dyes have simply to be fixed subsequently in a solvent vapour tank. The water flash-off, soaping, rinsing, and drying stages are the same as those employed for 100% acetate, triacetate, or Terylene goods. The complete dyeing range employed, therefore, differs in no respect from that required for dyeing the 100% goods.

DISCUSSION

The results achieved indicate quite clearly that the Vapocol method of dyeing offers the prospect of a very effective continuous dyeing technique for secondary cellulose acetate, cellulose triacetate, and, to a limited extent, Terylene fabrics. Also, making use of the Procion dyes, it can be used to dye blends with cellulosic fibres.

The main attractions of the method are the short dyeing times employed, which make for more compact equipment, the uniformity of treatment from side to side and from end to end of a fabric, and the manner in which the solvent vapour can be controlled. This last point is important in considering the cost of the plant, since the vapour tank and the water flash-off bath can be constructed at a relatively low cost either of galvanised iron plate or of stainless steel.

From the operating point of view the cost of the solvent is low because of the high recovery. Dye costs will in no case exceed those of conventional methods, and, in the case of triacetate fabrics, may be very significantly less for certain dyes, particularly in heavy depths, by reason of the higher tinctorial yield.

Provided that precautions are taken in designing the plant to limit the build-up of fabric tension in the vapour tank, no damage to the cloth structure results during dyeing. In this connection, a feature of the treatment which offers distinct attractions is that any creases in the fabric prior to dyeing are completely removed under tension in the trichloroethylene vapour.

In conclusion it should be pointed out that experience to date has been limited to pilot-plant operation, handling fabrics up to 12 in. in width, and that the design features of the equipment described in this paper are not necessarily applicable directly to bulk-scale machinery.

* * *

The author wishes to express his gratitude to Mr. A. Morrison, who has carried out the major part of the practical work, to Mr. J. Williamson, who drew up the design of the continuous plant, and to Mr. H. J. Ross and Mr. E. Stone of Imperial Chemical Industries Ltd., General Chemicals Division, without whose experience of handling fabric in trichloroethylene vapour the extension of the work to continuous processing would not have been possible.

IMPERIAL CHEMICAL INDUSTRIES LTD.
DYESTUFFS DIVISION
HEXAGON HOUSE
MANCHESTER 9

(*MS. received 15th October 1956*)

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## Discussion

MIDLANDS SECTION

Mr. J. WARDLE: Has any attempt been made to apply dyes of other classes than disperse dyes, i.e. of larger molecular size?

Mr. GARRETT: Attempts to apply dyes of large molecular size, such as pigment vat dyes, by the Vapocol process have not been successful. This may be due to the insolubility of the dyes in trichloroethylene or to the larger molecular size preventing penetration of the dyes into the fibres. The only other class of dyes apart from the disperse dyes which has been applied successfully has been the azoic, by the modified azoic technique, the fixation in trichloroethylene vapour being followed by the normal diazotisation in sodium nitrite and hydrochloric acid.

Mr. A. W. CARPENTER: Would the process offer a solution to some of the difficulties incurred in dyeing wool–Terylene blends?

Mr. GARRETT: Disperse dyes can be applied to Terylene fibres blended with wool by the method described, the degree of staining of the wool fibres being much less than that occurring in aqueous dyebaths. It seems likely that such staining as does occur by the Vapocol process takes place largely in the subsequent water treatment. The reasons why this application has not been fully investigated are that the length of wool–Terylene fabrics to be dyed to a single colour is generally too small to warrant the use of a continuous process and because of the limited range of colours which can be applied to Terylene in trichloroethylene vapour.

Mr. H. BELLIS: Are dark colours possible or is there some difficulty in padding or some other part of the process?

Mr. GARRETT: Dark colours, including full blacks, can be readily produced in dyeing times of 60 sec. on acetate and triacetate fabrics by the Vapocol process, using either direct or developed dyes.

Mr. W. L. LANGTON: There is a certain demand for a check fabric made from nylon and Terylene in which we are required to dye the former and reserve the latter. At some later date we may be required to dye the Terylene with a nylon reserve. This is impossible by conventional methods. Does this process completely reserve the nylon?

Mr. GARRETT: The fixation of disperse dyes on nylon in trichloroethylene vapour is very slight, and, as in the case of wool, such fixation as does occur probably takes place in the water bath.

Whilst a complete reserve of nylon would not be obtained, therefore, the degree of staining would be low and could readily be removed in a mild reduction bath.

Mr. B. D. HORNE: Manufacturers of cellulose triacetate do not recommend the use of trichloroethylene for dry cleaning. Has the lecturer any comments?

Mr. GARRETT: Trichloroethylene should not be used for the dry cleaning of triacetate fabrics because, even at room temperature, some diffusion of dye from the fibres takes place. This trouble is not encountered with perchlorethylene, which should be used for dry-cleaning purposes on this fibre.

Mr. D. O. DOUGLAS: Some years ago, attempts were made to apply vat acids from alcohol. Have any trials been made with this class of dye?

Mr. GARRETT: Attempts have been made to apply the vat acids by the Vapocol process, but no success has been achieved.

Mr. A. C. MILLS: Is there not a danger of the colours of nearby goods being affected by decomposition products from the trichloroethylene?

Mr. GARRETT: Provided that adequate precautions are taken to limit the escape of trichloroethylene from the plant, the concentration in the surrounding atmosphere would be so low that there would be no danger of damage to colours of nearby goods caused by decomposition products even if slight decomposition did occur.

Mr. J. RAYMENT: Has any work been done on set triacetate?

Mr. GARRETT: The exhaustion of disperse dyes on triacetate in the Vapocol process is virtually unaffected by any setting treatment carried out prior to dyeing. What is noticed is that triacetate fabrics which have been extended during the setting treatment shrink again on exposure to trichloroethylene vapour.

Mr. RAYMENT: Does this loss of width mean that the set has been destroyed?

Mr. GARRETT: The fabrics would have to be reset after the vapour treatment, though their stability may not necessarily have been destroyed.

Mr. MILLS: Is there any danger of roller marking due to the high degree of swelling rendering cellulose acetate more prone to damage?

Mr. GARRETT: No trouble has been encountered from this cause on any of the fabrics processed.

A MEMBER: Would there be any advantage in preheating Terylene fabrics to avoid the surface whiteness described?

Mr. GARRETT: Experiments carried out along these lines have not resulted in the elimination of the stripping of the surface hairs. The exhaustion of dyes has been found to decrease slightly if the fabric is preheated.

Mr. BELLIS: Are short runs a great disadvantage as in other continuous processes?

Mr. GARRETT: The minimum economic run has still to be determined by experience with full-scale plant. It is assumed that the labour of formulating a padding recipe and cleaning the pad range after dyeing one colour would tend to reduce the attraction of the Vapocol method for very short runs.

Mr. A. ARMITAGE: In the event of the padded-and-dried fabrics having an uneven moisture content face to face or selvedge to selvedge, would this produce unlevel dyeing after passing through the trichloroethylene developing bath?

Mr. GARRETT: No precise data are available on this point, but clearly variations in moisture regain will result in uneven condensation of trichloroethylene on the fabric. This would be unlikely to give rise to marked unlevelness in the case of those dyes which have a high exhaustion, but difficulties might be encountered with dyes which exhaust to only a limited extent. Any face-to-face variations of this sort would be partially levelled in the solvent tank, but selvedge-to-selvedge differences would not be corrected. If uniform drying of the fabric cannot be ensured, the precaution should be taken of reducing the moisture content of the fabric to the lowest possible figure.

A MEMBER: Is perchlorethylene of any use in the dyeing of Terylene?

Mr. GARRETT: The effectiveness of perchlorethylene vapour is approximately equal to that of trichloroethylene.

A MEMBER: Are there any risks of excessive condensation on the material in a trichloroethylene vapour bath?

Mr. GARRETT: If fabrics are immersed in the vapour for an extended time excess condensation may occur, but in the case of all the fabrics examined so far, exhaustion of dyes has been completed in a shorter time.

BRADFORD JUNIOR BRANCH

Dr. C. EARLAND: In view of the highly swollen state of the fibres in the Vapocol process, would not the ultimate uptake of dye be increased?

Mr. GARRETT: The evidence provided by the superior build-up of certain dyes on triacetate fibres suggests that the ultimate uptake of dye is in fact increased, but no attempts have been made to establish the limits of build-up quantitatively. No difficulty has been experienced in achieving the heaviest dyeings required on both secondary acetate and triacetate fabrics.

Mr. C. H. RUSHTON: Has trichloroethylene any degrading action on the disperse dyes?

Mr. GARRETT: No degradation occurs in the trichloroethylene vapour.

MANCHESTER SECTION

Professor R. H. PETERS: Does the fixation time require increasing to produce heavy depths?

Mr. GARRETT: The dyeing times quoted in Table III are sufficient to ensure fixation of heavy depths.

Mr. E. E. BOWKER: Does any levelling take place in the fixation process?

Mr. GARRETT: Levelling over shorter distances does take place in the trichloroethylene vapour bath, but the high exhaustion and rate of fixation prevent any widespread levelling.

Mr. C. NEWTON: What is the method recommended for matching a colour?

Mr. GARRETT: Laboratory matchings can be carried out using a small mangle of known expres-

sion and a small solvent vapour tank. The expression of the bulk-scale pad-mangle then has to be taken into account in determining the final recipe, but the conditions in the laboratory vapour tank are identical with those in large-scale plants, since in each case the solvent vapour is saturated and at the temperature of the boiling liquid.

Mr. J. W. REIDY: What is the reproducibility of the process?

Mr. GARRETT: This depends entirely upon the reproducibility of the padding process, since the conditions in the fixation bath are always identical.

Mr. F. W. BRADLEY: Does the particle size of the disperse dyes used affect the result?

Mr. GARRETT: Since disperse dyes have quite a marked solubility in the trichloroethylene liquid, the particle size required is not critical.

Mr. F. PARROTT: Is there any tendency to ending when a tertiary shade is produced by the Vapocol process?

Mr. GARRETT: The padding operation is generally carried out at a low temperature so that no affinity effects are observed. Provided that the nip pressure is maintained constant, there is no tendency for ending to occur at this stage, and since disperse dyes are compatible in the trichloroethylene fixation treatment, no difficulties are encountered in this part of the process.

Mr. MOORHOUSE: What are the conditions necessary to achieve satisfactory padding of hydrophobic fabrics?

Mr. GARRETT: We have found a single nip with two soft bowls operating under moderate pressure to give the most satisfactory results. Nafka gum thickener equivalent to 1–2% solids and a wetting agent are also required, and under these conditions padding can generally be carried out cold. Should difficulty be encountered in wetting out the fabric, raising the temperature of the pad liquor to 40°C. may be necessary. The wet fabric leaving the nip should be maintained under as high a uniform tension as possible across its width and, after passing over a minimum number of compensator rollers, should be dried in open width in a stenter or flue dryer providing uniform face-to-face drying.

Mr. NEWTON: Does the process affect the sublimation fastness of the dyes used?

Mr. GARRETT: The sublimation and all other fastness properties of fabrics dyed by the Vapocol process are equal to those of fabrics dyed by the conventional technique.

Mr. B. KRAMRISCH: Can the process be adapted to include the dyeing of yarn?

Mr. GARRETT: Provided that the yarn can be satisfactorily impregnated with dyes in a padding process, the fixation of the dye can be successfully carried out in trichloroethylene vapour. For single-end dyeing it is unlikely that this would be economic, but the process may have some application in the dyeing of warp beams.

Mr. N. HAMER: In a large unit, suitable for commercial use, would the loss of solvent be appreciably greater than that experienced on the experimental machine?

Mr. GARRETT: The principle of controlling the vapour by means of condensing coils can be applied successfully to tanks certainly up to 6 ft. in width, which would probably represent the maximum required for textile applications.

Mr. C. P. ATKINSON: I would confirm that large tanks can be used on a commercial scale with little loss of solvent. With regard to padding, I can confirm Mr. Garrett's statement that two soft bowls are to be preferred.

Mr. R. L. DERBY: Is there a real advantage in using thickeners in the padding process? What is their function?

Mr. GARRETT: Apart from the greater ease of padding generally rendered possible by the use of a thickener, it is necessary to use Nafka gum thickener in order to assist the removal of unfixed dye following the fixation process.

Mr. T. FLANAGAN: Can the process be extended to printing?

Mr. GARRETT: Because some levelling takes place in the trichloroethylene vapour, some bleed-out takes place from the edge of prints. This bleed-out can be prevented by preheating the fabric so as to limit the condensation of solvent vapour, but no investigation has been carried out into the practical application to print development.

Mr. FLANAGAN: What degree of staining of viscose rayon is obtained using the Vapocol process compared with dyeing in a conventional dyebath?

Mr. GARRETT: The degree of staining of viscose rayon is of the same order as that occurring in conventional dyebaths.

Mr. CLARK: With the Procion–Vapocol technique is there not a large wastage of dye?

Mr. GARRETT: Considerable wastage of dye may occur in fabrics in which one fibre component occurs in, say, warp or weft bars, the dye padded on between the bars being wasted. In the case of brocade fabrics and blend fabrics, in which an overall coverage of both fibre components is present, migration of Procion and disperse dyes to their appropriate fibres takes place, and so the wastage is largely eliminated.

Mr. L. MORRIS: Which thickeners do you recommend for dyeing cellulose acetate–viscose rayon blends by the Procion–Vapocol technique?

Mr. GARRETT: Nafka gum thickener is used in order to secure rapid and complete removal of unfixed dye in the soaping baths.

Mr. ATKINSON: In this process there are three drying stages. Can any way be foreseen of eliminating any of these?

Mr. GARRETT: The three drying stages referred to are those following scouring, padding, and the final soaping baths. Of these the only one which could possibly be eliminated would be that following the scouring if, instead, loom-state fabric were padded directly. This could possibly be achieved, provided that no dirt were present on the fabrics, if the size on the loom-state fabric could be readily wetted out in the pad liquor, easily penetrated by the trichloroethylene vapour, and readily removed in the final soaping bath. This technique has been successfully carried out with a number of fabrics, but its success may depend upon the nature of the size employed.

COMMUNICATIONS

# Determination of the Migratory Properties of Direct Dyes

JOSÉ CEGARRA

The effects have been studied of four variables— temperature, electrolyte concentration, liquor ratio, and agitation— on the migration of direct dyes selected from Classes A, B, and C. At low temperatures increase of temperature has a more favourable influence with Classes A and B than with Class C. An optimum electrolyte concentration for maximum migration is shown by dyes of Classes A and B, but with Class C the migration diminishes steadily as the electrolyte concentration is increased. Increase in liquor ratio increases migration of B and C dyes, but not that of A dyes. Agitation increases migration in all cases.

## Introduction

The main factors determining the levelling power of a dye are "strike" and migration. The former was considered in an earlier paper [1], and the object of the work described here was to investigate the migration of direct dyes on cotton. Three dyes were chosen, representing Classes A, B, and C respectively [2], and their migration properties under different dyeing conditions were studied.

Two main methods are available for determining the migration properties of a dye [3]—

(*a*) The time that two patterns dyed in the same bath require to reach the same colour, one being entered a constant interval of time after the other, generally 5 min. for direct dyes.

(*b*) Finding the quantities of dye transmitted from a dyed pattern in a blank dyebath to an undyed pattern during different intervals of time.

The method adopted in the experiments described here was the second one, since it allowed the process to be studied on a quantitative basis, whereas the first method depends on a subjective judgment.

With this second method, some preliminary equilibrium dyeings must be carried out with the dye to be studied. The dyed pattern and an undyed pattern of the same weight are introduced into a blank dyebath set according to the variable under investigation. Dyeing is continued for a given time, and then the quantity of dye remaining on the original dyed pattern, the quantity that has migrated to the undyed pattern, and the quantity that remains in the residual bath are determined. Every such experiment gives a point on a graph, and a curve drawn through such points gives a graphical representation of the migration of the dye, the ordinates showing the amounts of dye on the two patterns after the times of "dyeing" shown by the abscissae.

## Experimental

### 1. PURIFICATION OF THE DYES

The commercial samples supplied by Imperial Chemical Industries Ltd. were purified by Robinson and Mills' method [4], which was modified somewhat for Chlorazol Sky Blue FF on account of its high solubility [5]. The purified dyes gave the following analyses—

| | |
|---|---|
| Chrysophenine G (C.I. Direct Yellow 12) ... | 98·7% |
| Chlorazol Sky Blue FF (C.I. Direct Blue 1) ... | 98·1% |
| Chlorazol Azurine G (C.I. Direct Blue 8) ... | 98·3% |

### 2. PRELIMINARY DYEINGS

For the preliminary dyeings cotton fabric was used weighing 104 g./sq.metre, previously scoured and bleached. This fabric was cut into strips each weighing 11 g., sufficient to give after dyeing five 2-g. patterns. The dyeing conditions were as follows—

| | |
|---|---|
| Dye ... ... | 0·5% on weight of dry cotton |
| Sodium chloride ... | 10% on weight of dry cotton |
| Liquor ratio ... | 30 : 1 |
| Temperature ... | 100°c. |
| Time of dyeing ... | 90 min. |

When the strips had been dyed and dried, they were cut into five equal strips, each of which served to determine a point on the curve of the migration test.

### 3. MIGRATION TESTS

Each group of five patterns from the same 11-g. strip was taken together with five undyed patterns of the same fabric of approximately the same weight as the dyed patterns. Every dyed pattern and the corresponding undyed pattern served to determine a point on the curve representing a single experiment, the times of contact of the five pairs of patterns being 10, 20, 30, 60, and 90 min. respectively.

TABLE I

| Variable | Temp. (°c.) | Electrolyte (NaCl) Concn. | Liquor Ratio | Agitation (r.p.m.) |
|---|---|---|---|---|
| (1) Temperature ... | 30, 60, 80, 100 | 10%* | 20 : 1 | 160 |
| (2) Electrolyte concn. ... | 100 | 0, 20, 40, 60% | 20 : 1 | 160 |
| (3) Liquor ratio ... | 100 | 5 g./litre | 10, 20, 40, 60 : 1 | 160 |
| (4) Agitation ... ... | 100 | 10% | 20 : 1 | 0, 80, 120, 160 |

*On weight of dry fabric

Table I shows the conditions under which the various factors influencing migration have been investigated.

### 4. APPARATUS

The migration tests were carried out in a specially designed apparatus. The temperature of the heater could be automatically controlled to ±1°c., and the speed of stirring could be varied between 60 and 200 r.p.m. The containers consisted of two glass tubes joined at the middle, one for the dyed pattern and one for the undyed pattern. In order to keep the volume of the bath constant, both tubes were provided with interchangeable condensers. The patterns were held on stainless-steel supports, which could be rotated by the stirring mechanism, allowing the patterns to move through the blank dyebath.

With this apparatus five pairs of patterns can be studied independently at the same time, provided that they are all at the same temperature.

### 5. DETERMINATION OF DYE DISTRIBUTION

The quantity of dye remaining in the blank dyebath after the migration test is determined colorimetrically by means of a Hilger Spekker absorptiometer type H 760.

The quantity of dye absorbed by the undyed pattern and that retained by the dyed pattern are determined by extraction with a 20% solution of pyridine in a Soxhlet followed by colorimetric estimation. In the case of Chlorazol Azurine G, which, unlike the other two dyes, undergoes appreciable alteration in colour in contact with the hot pyridine solution, it is necessary to plot a calibration curve under the same conditions as the extractions of the dye, so as to avoid errors in the determinations.

## Results

The results are all plotted as percentages of the initial content finally distributed among the two patterns and the blank dyebath: in this way all curves for the dyed patterns start at the same level (100%), and no error of interpretation is introduced in view of the very slight differences in weight of dye absorbed by a given weight of fibre in the different patterns. The curves thus obtained (Fig. 1–17) show clearly the behaviour of the dyes under the different conditions tested. It must be emphasised, however, that no experimental points were determined in the time interval 0–10 min., so that the curves here are purely hypothetical.

On the basis of these curves, the influence of the various factors investigated may be summarised as follows.

### 1. TEMPERATURE

As can be seen from Fig. 1–3, migration increases as the temperature rises, but the rate of increase diminishes in the order: Chrysophenine G > Chlorazol Sky Blue FF > Chlorazol Azurine G. Thus the conditions for a comparable migration, e.g. for a difference between the ordinates for the dyed and undyed patterns of 50% of the initial dye content, are shown in Table II.

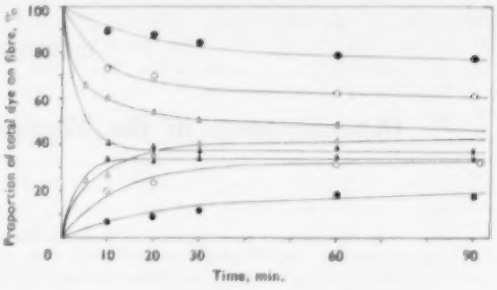


FIG. 1

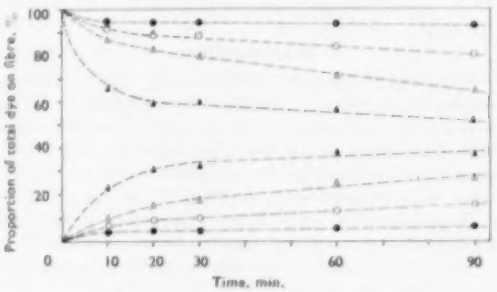


FIG. 2

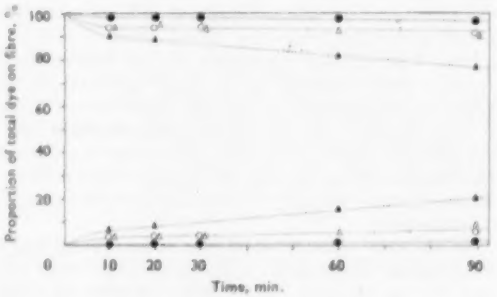


FIG. 3

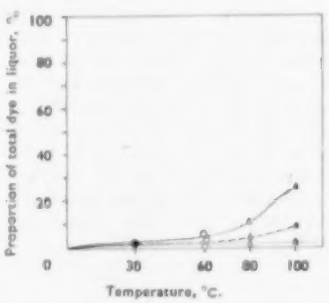


FIG. 4

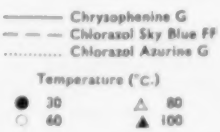


FIG. 1–4— Effect of Temperature on Migration

TABLE II

| Dye | *Colour Index* No. | Temp. (°c.) | Time (min.) |
|---|---|---|---|
| Chrysophenine G | C.I. Direct Yellow 12 | 60 | 13 |
| Chlorazol Sky Blue FF | C.I. Direct Blue 1 | 80 | 56 |
| Chlorazol Azurine G | C.I. Direct Blue 8 | 100 | 107 |

The quantity of dye that remains in the blank dyebath also increases with temperature in the same order (Fig. 4). Thus for 100°c. and 90 min. the ordinates for the residual bath are 26, 9, and 2·5% for the three dyes respectively. We can obtain an idea of the effect of the dye in the bath on the depth of colour of the two patterns by observing the displacement of the "axis of symmetry" (or line of mean values) of the desorption and absorption curves toward the zero axis as the temperature is raised (Table III).

Fig. 1 shows that with Chrysophenine G and dyes of similar behaviour at 100°c. migration reaches an equilibrium state within 20 min., for the slight difference in depth shown by the curves is not visible to the eye; at 80°c. equilibrium is established after 40 min., and the difference in colour between the two patterns is more marked.

TABLE III

**Mean Values (%) of Desorption and Absorption Curves**

| Temperature, °c. ... ... ... ... ... | 30 | 60 | 80 | 100 |
|---|---|---|---|---|
| Chrysophenine G C.I. Direct Yellow 12 ... | 48·5 | 47·5 | 44·5 | **37·0** |
| Chlorazol Sky Blue FF C.I. Direct Blue 1 ... ... | 49·2 | 48·7 | 48·0 | 45·5 |
| Chlorazol Azurine G C.I. Direct Blue 8 ... ... | 49·5 | 49·2 | 49·0 | **48·7** |

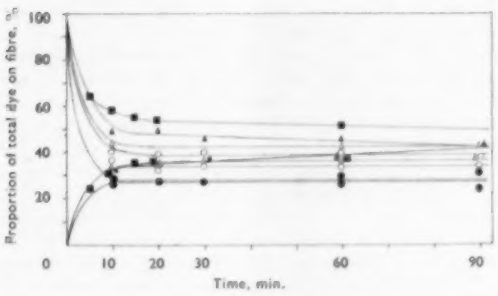

FIG. 5

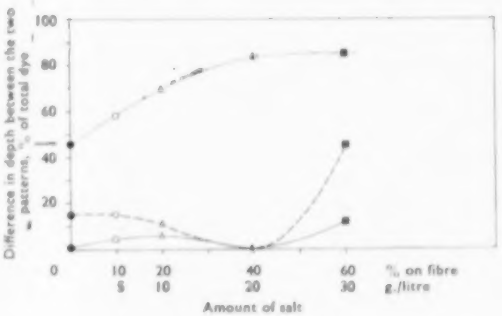

FIG. 8

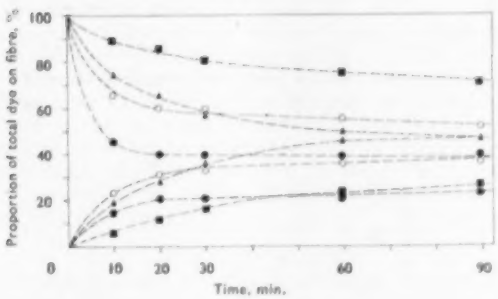

FIG. 6

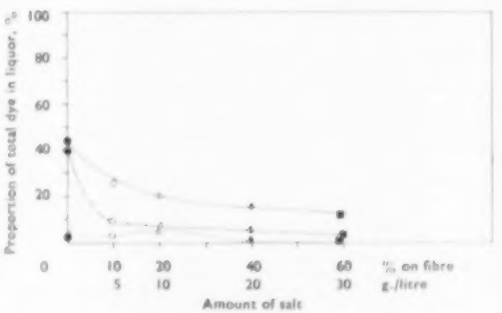

FIG. 9

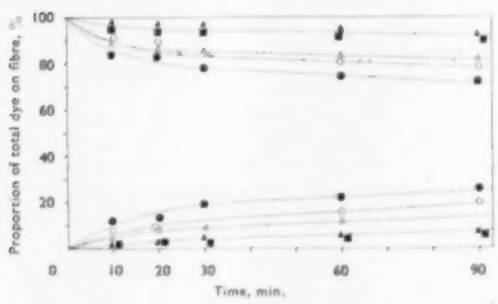

FIG. 7

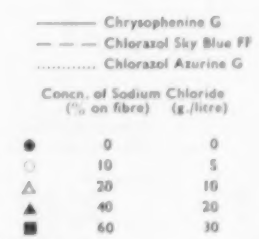


FIG. 5–9— Effect of Salt Concentration on Migration

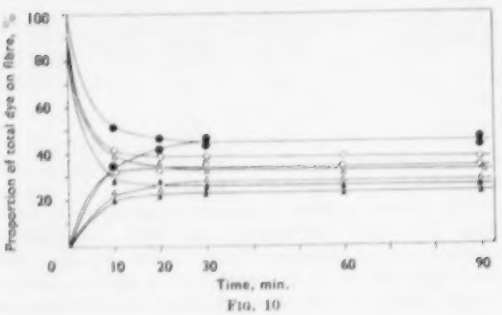

FIG. 10

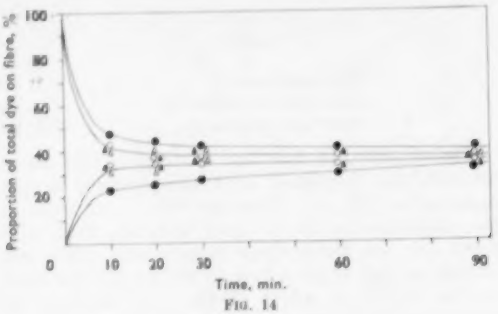

FIG. 14

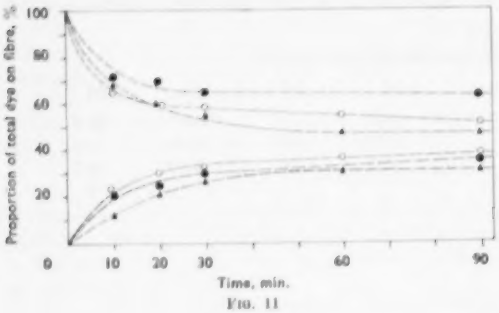

FIG. 11

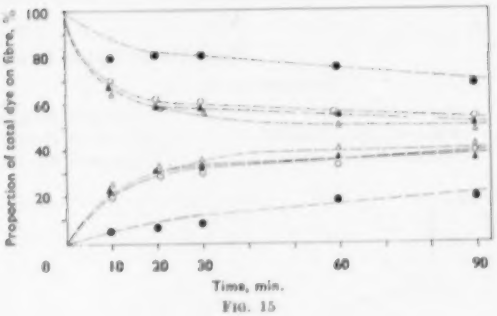

FIG. 15

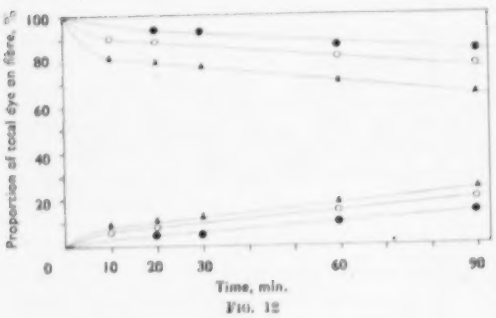

FIG. 12

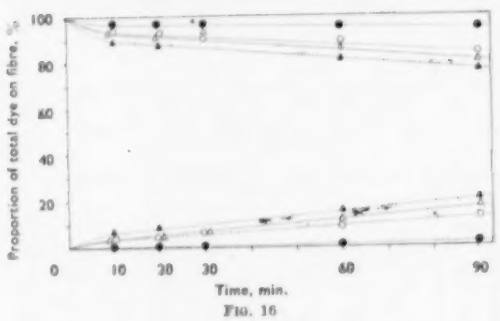

FIG. 16

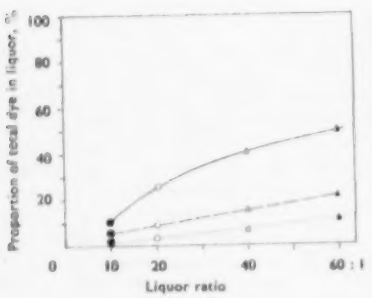

FIG. 13

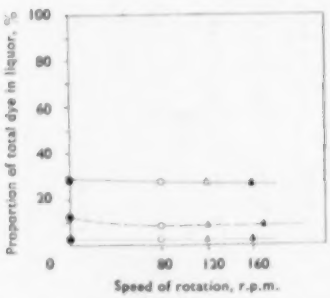

FIG. 17

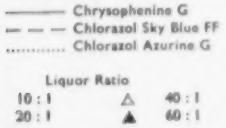


FIG. 10–13— Effect of Liquor Ratio on Migration

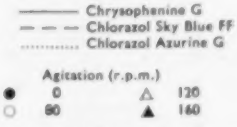


FIG. 14–17— Effect of Agitation on Migration

TABLE IV

**Mean Values (%) of Desorption and Absorption Curves**

| Salt, % ... ... ... ... | 0 | 10 | 20 | 40 | 60 |
|---|---|---|---|---|---|
| Chrysophenine G C.I. Direct Yellow 12 ... | 28·0 | 37·0 | 39·5 | 42·0 | 44·0 |
| Chlorazol Sky Blue FF C.I. Direct Blue 1 ... ... | 30·0 | 45·5 | 46·5 | 47·5 | 48·5 |

With Chlorazol Sky Blue FF and similar dyes (Fig. 2) at temperatures below 100°C. equilibrium is not reached within the intervals of time allowed for these tests, although we may suppose that at 100°C. itself the time required for equilibrium is very close to 120 min. It may be hoped that at temperatures above 100°C. the migration equilibrium will be reached in shorter times. In the case of Chlorazol Azurine G the form of the migration curves makes it difficult to express any views on the attainment of equilibrium.

### 2. ELECTROLYTE CONCENTRATION

As in the case of temperature, electrolyte concentration has a general effect on migration, while the different dyes examined also show special features (Fig. 5–8).

With Chrysophenine G migration to the undyed fibre is greatest in the absence of electrolyte, and steadily decreases as the electrolyte concentration increases; it passes through a minimum, and reaches a maximum again (40% salt at 90 min.), after which it falls again (Fig. 8). Chlorazol Sky Blue FF shows the same optimum salt concentration (40% at 90 min.), although its behaviour in absence of salt is different.

The behaviour of these two dyes is similar with respect to the residual bath, the strength of which decreases as the electrolyte concentration increases, and is greater with Chrysophenine G than with Chlorazol Sky Blue FF. Table IV confirms that the mean values of the desorption and absorption curves increase with increase in salt concentration.

The behaviour of Chlorazol Azurine G is different from that of the other two dyes, for it does not show an optimum salt concentration, but in general migration decreases as the quantity of electrolyte increases. No conclusion can be drawn from its behaviour in the residual bath, possibly because of experimental error.

### 3. LIQUOR RATIO

As can be seen from Fig. 10–13 the behaviour of Chrysophenine G differs from that of the other two dyes, for while with the latter an increase in liquor ratio increases the migration, the difference in depth between the two patterns tending to lessen, with Chrysophenine G optimum conditions are obtained when the liquor ratio is a minimum.

The behaviour of the residual bath is similar with all three dyes, its strength tending to increase as the liquor ratio increases, so that the equilibrium depths of both patterns tend to be lower. This loss of depth is most marked with Chrysophenine G.

### 4. AGITATION

In all cases migration is increased by agitation of the patterns in the bath (Fig. 14–16). The rate of agitation has little effect, which is rather more marked with Chlorazol Azurine G (Fig. 16). The slight anomalies evident in Fig. 15 are attributable to experimental error. From Fig. 17 it appears that the amount of dye in the residual liquor falls very slightly with increasing rate of agitation.

## Discussion

If we analyse the behaviour of the dye during the occurrence of migration, from the dyed pattern through the aqueous solution to the undyed pattern, we can distinguish two well defined phases— the kinetic and the equilibrium stages. During the first the dye is desorbed from the dyed pattern, more or less strongly according to the conditions and the nature of the bath. This produces a concentration of the dye in the bath, leading to absorption by the undyed pattern, which is controlled by the conditions of the dyeing system. The desorption of the dye will be controlled by its affinity for the fibre and by the ease with which it can diffuse through the fibre to reach the aqueous solution. Thus dyes of high affinity and low diffusibility will undergo a slow desorption, but once they are in the bath they will be taken up rapidly by the undyed fibre, and then diffuse slowly into the interior of the fibre. In such cases the establishment of equilibrium between the dyed fibre, the bath, and the undyed fibre will be slow and will be characterised by the small quantity of dye that will be present in the aqueous solution during the experiment. On the other hand, dyes of low affinity and high rate of diffusion are characterised by rapid establishment of equilibrium and a high concentration of dye in the bath. Equilibrium will be reached when the concentrations of dye in the two patterns and in the aqueous solution remain unchanged even when the time of contact is prolonged indefinitely; the concentrations of dye in the two patterns will then be identical.

On the basis of the behaviour of the above two types of dye in the kinetic and equilibrium stages, therefore, an attempt will be made to interpret the observed effects of the various factors on migration.

### 1. TEMPERATURE

The effect of temperature on the kinetic stage of migration is to increase the rate of desorption [6, 7], in accordance with its known effect on rate of dyeing. In the author's view this is a reflection of the increase in the diffusion coefficient, which is more or less opposed by the affinity of the dye for the fibre. This can be fully appreciated from the results plotted in Fig. 1–4, which show that, when the affinity is high and the coefficient of diffusion is low (Chlorazol Azurine G), the migration of the dye is very small, with very little dye in the bath in consequence of its high affinity. Chrysophenine G shows the opposite behaviour, for it quickly reaches the equilibrium stage and a large quantity of dye remains in the solution. When the system arrives

at equilibrium, its behaviour is identical with that of normal dyeings, there being more dye on the two patterns and less in the residual bath at low than at high temperatures.

It must be emphasised that, for dyes like Chrysophenine G, the use of temperatures higher than 100°c. will produce no improvement in migration from the industrial point of view, for at this temperature equilibrium is reached in 15 min. in the system employed; also, the greater the decrease in the exhaustion, the greater will be the time required to lower the bath temperature in order to increase the value of the exhaustion. For the other dyes the effect of a temperature greater than 100°c. is of great importance for obtaining perfect migration, the temperature required being higher for Chlorazol Azurine G than for Chlorazol Sky Blue FF.

### 2. ELECTROLYTE CONCENTRATION

The varying effect of electrolyte on different dyes, and the resemblances between Chrysophenine G and Chlorazol Sky Blue FF in contrast to Chlorazol Azurine G, can be explained on the basis of studies made by Neale and Stringfellow [8], Boulton and Morton [9], and Crank [10, 11] on the influence of electrolyte on the diffusion coefficient and its theoretical interpretation. According to Crank the apparent diffusion coefficient $D_a$ depends on the Donnan distribution coefficient $\lambda$ and on the ratio of the equilibrium dye concentrations in the bath and in the fibre—

$$D_a = k\lambda^z \frac{[D]_s}{[D]_f}$$

where $k$ is a constant and $z$ is the valency of the diffusing ion.

When the electrolyte concentration in the migration bath is zero, $\lambda$ will be very small, and the value of $\lambda^z$ will be still smaller the larger is the valency of the ion. In the present case the minimum value of $\lambda^z$ corresponds to Chlorazol Sky Blue FF with $z = 4$, and the maximum values correspond to $z = 2$ for the other two dyes. The effect of $[D]_s/[D]_f$ depends on the affinity of the dye for the fibre, which, in the absence of salt, is different for the three dyes. It can be seen from Fig. 9 that this ratio is very high for Chrysophenine G and Chlorazol Sky Blue FF but quite low for Chlorazol Azurine G. In consequence, we find that under the above conditions, because of the predominance of $[D]_s/[D]_f$ over $\lambda^z$, the value of $D_a$ is greatest for Chrysophenine G, smaller for Chlorazol Sky Blue FF, and least for Chlorazol Azurine G.

As the concentration of electrolyte increases, the value of $\lambda$ increases rapidly, $\lambda^4$ for Chlorazol Sky Blue FF increasing still more rapidly than $\lambda^2$ for the other two dyes, until at greater concentrations $\lambda$ becomes constant at a value approaching unity. The factor $[D]_s/[D]_f$ decreases as the quantity of electrolyte increases, so that for $D_a$, as the product of two antagonistic factors, the following cases can occur—

(*a*) $\Delta\lambda^z > -\Delta\left(\frac{[D]_s}{[D]_f}\right)$

(*b*) $\Delta\lambda^z = -\Delta\left(\frac{[D]_s}{[D]_f}\right)$

(*c*) $\Delta\lambda^z < -\Delta\left(\frac{[D]_s}{[D]_f}\right)$

(*d*) $\lambda^z$ constant and $\Delta\left(\frac{[D]_s}{[D]_f}\right)$ decreasing.

It appears that Chlorazol Sky Blue FF and Chrysophenine G behave according to (*a*) and subsequently according to (*d*), while for Chlorazol Azurine G condition (*c*) is followed by (*d*), which would explain the effect of electrolyte concentration on the migration of these dyes. Nevertheless, the number of measurements made so far is insufficient to draw definite conclusions on the disparity of behaviour of direct dyes in migration, and further experiments are necessary to confirm or disprove the above hypothesis.

### 3. LIQUOR RATIO

The influence of this factor is different according to whether the dyed or the undyed pattern is considered, and also depends on the type of dye used. If the dye has little affinity for the fibre, a low liquor ratio is sufficient for desorption to occur, so that the concentration of dye in the bath rises and dye is rapidly absorbed by the undyed fibre. As the liquor ratio is increased, desorption becomes more marked, but the rate of dyeing of the undyed pattern is lower. Finally, a state of equilibrium is reached, in which the effect of increasing liquor ratio is to reduce the amount of dye on both fibres (Fig. 10), as happens in the normal dyeing process. With dyes such as Chlorazol Azurine G, also, migration of the dye into the bath increases with increasing liquor ratio, but the high affinity opposes the effect of dilution of the bath, so that a greater quantity of dye is absorbed by the undyed pattern (Fig. 12). At equilibrium the effect is the same as in the previous case.

### 4. AGITATION

It is to be expected that the influence of this factor will be evident only during the kinetic period of migration, and that it will be favourable (Fig. 14–16). Nevertheless, when the temperature and other conditions bring the system to equilibrium very quickly, the effect of an increase in agitation is practically negligible (Fig. 14).

## Conclusions

The following conclusions may be drawn from the above results and discussion—

(*a*) Increase in temperature produces a marked increase in the migration of the dyes investigated, which depends on the affinity of the dye and on the increase of its diffusion coefficient with temperature. With dyes like Chrysophenine G temperatures above 100°c. confer no practical advantage, but with the other dyes they bring the system to equilibrium more rapidly, provided that the dyes do not decompose at such temperatures.

(*b*) When equilibrium is attained, the concentrations of the dye on the originally dyed and undyed patterns are identical, their value in equilibrium with the dye concentration in the migration bath following the general law governing dyebath equilibrium as a function of temperature.

(*c*) The influence of electrolyte concentration on migration seems to be capable of interpretation on Crank's theory. But more data are required, for other dyes, before the results obtained with the three dyes investigated can be regarded as definitely established. The existence of an optimum concentration of electrolyte in the migration bath has been confirmed for Chrysophenine G and Chlorazol Sky Blue FF, but not for Chlorazol Azurine G.

(*d*) When the dye has little affinity for the fibre, liquor ratio has little effect on migration at favourable temperatures, but the concentration of dye on the fibre decreases as the volume of the bath increases. With dyes of high affinity, increase in liquor ratio favours migration, and the concentration of dye on the fibre at equilibrium falls relatively little.

(*e*) Agitation tends to increase migration, its effect being the greater the further the dyeing system is from equilibrium as a result of other factors.

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LABORATORIOS DE INVESTIGACIÓN TEXTIL
ESCUELA ESPECIAL DE INGENIEROS DE INDUSTRIAS TEXTILES
TARRASA
SPAIN

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# The Measurement of Donnan Potentials with Dyed Cellulose and Aqueous Salt Solutions

S. M. NEALE and P. K. SAHA

Measurements have been made of the electrical potential difference (Donnan potential) between dyed Cellophane and aqueous solutions of alkali-metal chlorides. The presence of adsorbed coloured ions of negative electric charge increases the negative potential of cellulose, whereas positively charged coloured ions may reverse the sign of the charge. The measured potentials, whilst smaller than those predicted by the application of the Donnan theory, after making the usual simplifying assumptions, are in good general agreement with modern views of the mechanism of direct dyeing.

It was shown by Neale and Standring [1] that the electrical potential difference between water-swollen cellulose and an aqueous solution can be ascribed to the presence of ionogenic groups (COOH) in the cellulose structure. The variation of the negative potential of the membrane with pH and with salt concentration was found to be in accord with the Donnan theory of membrane equilibrium.

When cellulose is dyed with direct dyes, it is generally agreed that the coloured ion alone is attached to the substrate by means of specific forces [2]. Consequently, when the adsorbed dye ion is negatively charged, the negative charge on the membrane structure (which will be called the "A value") and the Donnan potential are increased. On the other hand, with the adsorption of a positive dye ion, the charge and the potential of the membrane may be annulled or reversed [3].

According to the theory of Hanson, Neale, and Stringfellow [4] it should be possible to calculate the magnitude of the potential difference if the net electric charge and the effective volume of the swollen cellulose (regarded as an equipotential volume) are known. It will be shown that the results now presented are in substantial agreement with such a calculation.

The calculation is carried out as follows—

$[A^-]_0$ = Concentration of anions due to intrinsic COOH groups in cellulose [1]
= 0·018 g.-equiv./1000 g. water
$x$ = Concentration of dye absorbed by cellulose, g.-equiv./1000 g. cellulose
$[A^-]_1$ = Concentration of adsorbed anions
$= \frac{x}{1{\cdot}17}$ g.-equiv./1000 g. water
(since 1·17 g./g. = water imbibition of cellophane)
$[A^-]$ = Total value due to $-COO^-$ + adsorbed anions
$= 0{\cdot}018 + \frac{x}{1{\cdot}17}$

Then the Donnan potential between cellulose and a solution of any uniunivalent electrolyte of molality $m_2$ will be—

$$\psi_2 = \frac{RT}{F} \sinh^{-1}\frac{[A^-]}{2m_2}$$

assuming unit activity coefficients [1].

## EXPERIMENTAL METHODS

This work was carried out some years before that described by Neale and Standring [1], in which the Donnan potential of undyed Cellophane was examined over a wide range of salt concentrations, by using a series of electrochemical cells containing potassium chloride solutions at a concentration ratio of 2:1. In the present work the cells were set up with one solution saturated and the other dilute (Fig. 1), three alkali-metal chlorides MCl being used in turn. Then, if—

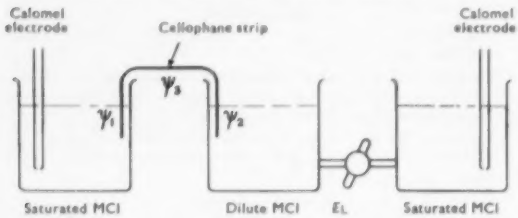



FIG. 1

$E$ = Observed e.m.f.
$\psi_1$ = p.d. between Cellophane strip and saturated MCl soln.
$\psi_2$ = p.d. between Cellophane strip and dilute MCl soln.
$\psi_3$ = Diffusion potential
$E_L$ = Liquid-liquid junction potential

$\psi_1$ may be taken as zero to a close approximation [1]. $E_L$ may be calculated from the following approximate equation—

$$E_L = \frac{RT}{F}(t_+ - t_-)\ln\frac{m_1}{m_2}$$

where $t_+$ and $t_-$ are the cationic and anionic transport numbers, and $m_1$ and $m_2$ are the molalities of the salt solutions. The value of $\psi_3$ is calculated from the expression derived by Meyer and Sievers [5]—

$$\psi_3 = -\frac{RT}{F}U\ln\frac{x_1 + [A^-]U}{x_2 + [A^-]U}$$

where—

$$U = \frac{u - v}{u + v}$$

$$x = \sqrt{[A^-]^2 + 4m^2}$$

The unknown potential $\psi_2$ is therefore obtained from—

$$\psi_2 = E + \psi_3 - E_L$$

The experimental set-up was very simple. Dilute potassium or other metal chloride solution was placed in one of the limbs of the Cambridge Morton glass-electrode assembly, connected through the tap junction to a saturated solution contained in the other limb. A small beaker also containing the saturated solution stood on a polythene block close to the limb containing the dilute solution. Two saturated calomel electrodes were placed in the two vessels containing saturated solutions (Fig. 1) and connected to a Fielden valve electrometer and a potentiometer sensitive to 0·1 mv. The connection between the saturated solution in the beaker and the dilute solution was made by means of a strip of Cellophane about 1 cm. wide, which had been soaked in the dilute solution and "blotted" with dry filter paper to remove surplus liquid.

The e.m.f. then recorded gave the total value $E$ for the system, although a small correction was usually required for asymmetry of potential between the two calomel electrodes, determined by placing them both in the same solution.

It is necessary to use an electrometer valve potentiometer, because the electrical resistance of the Cellophane strip may amount to several megohms.

So as to avoid possible zero errors in the potentiometer, and in order to increase the sensitivity of the measurements, the cell system was connected, via a reversing switch, first in series with and then in opposition to a Weston standard cell—

$$E = \tfrac{1}{2}[(E_S + E_1) - (E_S - E_2)]$$

The Cellophane samples used were of three different thicknesses and from two batches, designated O 300, O 400, N 300, N 400, and N 600 respectively. They were repeatedly washed with distilled water, and then air-dried, before use.

The dyed samples were prepared with two anionic dyes, Benzopurpurin 4B (C.I. Direct Red 2) and

TABLE I

**Donnan Potentials (mv.) of Undyed Cellophane N 600**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Molality of dilute soln. ... ... | | | | 0·1 | 0·05 | 0·02 | 0·01 | 0·001 |
| KCl | $E$ | (obs.*) ... ... | | 2·0 | 5·5 | 14·0 | 23·3 | 63·4 |
| (conc. soln. | $E_L$ | (calc.) ... ... | | 2·0 | 2·3 | 2·7 | 3·0 | 3·6 |
| saturated) | $\psi_3$ | (calc.) ... ... | | 2·0 | 2·4 | 2·8 | 3·1 | 3·4 |
| | $\psi_2$ | (exptl.†) ... ... | | 2·0 | 5·6 | 14·1 | 23·4 | 63·2 |
| $\psi_2$ by sheet method | | ... ... ... ... | | 3·0 | 6·1 | 11·3 | 21·0 | — |
| NaCl | $E$ | (obs.) ... ... | | 5·5 | 8·5 | 16·0 | 27·6 | 81·3 |
| (conc. soln. | $E_L$ | (calc.) ... ... | | 29·1 | 33·6 | 38·8 | 42·6 | 55·8 |
| saturated) | $\psi_3$ | (calc.) ... ... | | 25·7 | 29·9 | 34·9 | 37·8 | 40·3 |
| | $\psi_2$ | (exptl.) ... ... | | 2·1 | 4·8 | 12·1 | 22·8 | 65·5 |
| LiCl | $E$ | (obs.) ... ... | | 2·7 | 5·6 | 14·6 | 25·6 | — |
| (conc. soln. | $E_L$ | (calc.) ... ... | | 23·0 | 28·7 | 36·4 | 42·2 | — |
| molar) | $\psi_3$ | (calc.) ... ... | | 21·8 | 28·2 | 35·9 | 40·8 | — |
| | $\psi_2$ | (exptl.) ... ... | | 1·7 | 5·3 | 14·3 | 24·4 | — |
| For all salts | $\psi_2$ | (calc. from $[A^-] = 0·018$) | | 2·2 | 4·5 | 11·0 | 20·5 | 73·3 |
| | $\psi_2$ | (obs.[1] for KCl) | | 2·4 | 4·8 | 12·0 | 19·0 | 57·2 |

* Observed
† Experimental

TABLE II

**Donnan Potentials of Undyed Cellophane O 300**

| | Molality of dilute soln. | | | 0·1 | 0·05 | 0·02 | 0·01 | 0·001 |
|---|---|---|---|---|---|---|---|---|
| KCl | $E$ | (obs.) | ... ... | 3·0 | 6·0 | 16·5 | 26·1 | 60·8 |
| (conc. soln. | $E_L$ | (calc.) | ... ... | 2·0 | 2·3 | 2·7 | 3·0 | 3·6 |
| saturated) | $\psi_2$ | (calc.) | ... ... | 2·0 | 2·4 | 2·8 | 3·0 | 3·1 |
| | $\psi_2$ | (exptl.) | ... ... | 3·0 | 6·1 | 16·6 | 26·1 | 60·3 |
| NaCl | $E$ | (obs.) | ... ... | 6·0 | 9·5 | 20·0 | 31·0 | 79·3 |
| (conc. soln. | $E_L$ | (calc.) | ... ... | 29·1 | 33·6 | 38·8 | 42·6 | 55·8 |
| saturated) | $\psi_2$ | (calc.) | ... ... | 25·7 | 29·7 | 34·4 | 36·5 | 37·9 |
| | $\psi_2$ | (exptl.) | ... ... | 2·6 | 5·6 | 15·6 | 24·9 | 61·4 |
| LiCl | $E$ | (obs.) | ... ... | 3·8 | 7·9 | 18·0 | 32·5 | — |
| (conc. soln. | $E_L$ | (calc.) | ... ... | 23·0 | 28·7 | 36·4 | 42·2 | — |
| molar) | $\psi_2$ | (calc.) | ... ... | 21·7 | 27·2 | 34·6 | 37·4 | — |
| | $\psi_2$ | (exptl.) | ... ... | 2·7 | 6·6 | 14·3 | 25·6 | — |
| For all salts | $\psi_2$ | (calc. from $[A^-] = 0·018$) | ... | 2·2 | 4·5 | 11·0 | 20·5 | 73·3 |
| | $\psi_2$ | (obs.[1] for KCl) | | 2·4 | 4·8 | 12·0 | 19·0 | 57·2 |

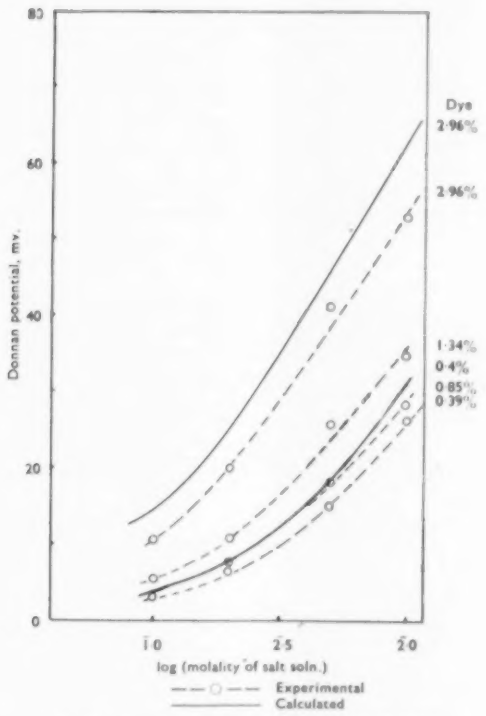



FIG. 2—Donnan Potentials of Cellophane dyed with C.I. Direct Blue 1

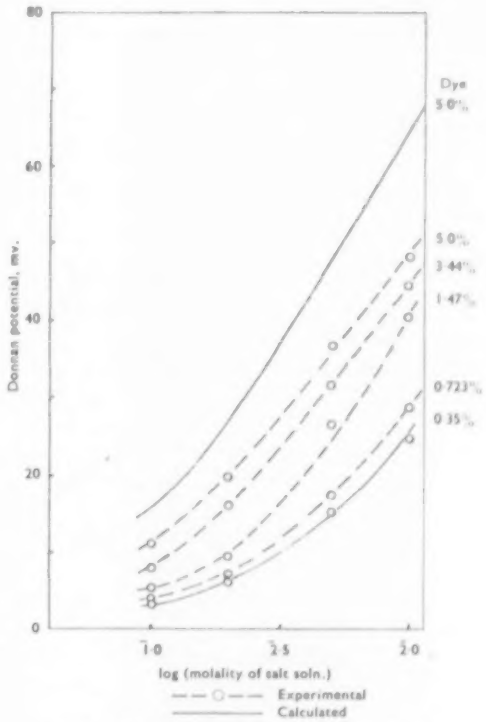



FIG. 3—Donnan Potentials of Cellophane dyed with C.I. Direct Red 2

Chlorazol Sky Blue FF (C.I. Direct Blue 1), as well as the cationic direct dye, Victoria Blue B (C.I. Basic Blue 26). The dyeings were carried out with purified dye samples at the boiling point for 4 hr. No salt was added to the Victoria Blue dye-baths, since, as this is a cationic dye, decreased absorption may result[6]. The dyed Cellophane was rinsed with ice-cold water and dried with filter paper, as before.

The dye content of the samples was estimated in the usual way by colorimetric analysis of "strippings" with 25% pyridine.

## EXPERIMENTAL RESULTS

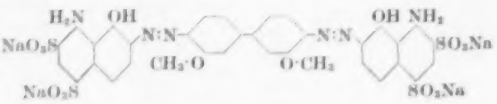
Results for two samples of Cellophane are given in Tables I and II. As the other three samples gave closely similar potentials, the values for these are not recorded here.

The row of figures in Table I corresponding to "$\psi_2$ by sheet method" was obtained by setting up the cell with twenty sheets of Cellophane clamped transversely, packed tightly together, between the two solutions, which were stirred mechanically. This was much more laborious than the "strip method", and as the numerical results were

TABLE III

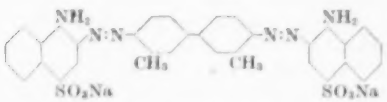
**Donnan Potentials (mv.) of Dyed Cellophane**

(In each pair of figures, the upper one is the experimental value and the lower one the calculated value)



C.I. Direct Blue 1
(mol. wt. 992)

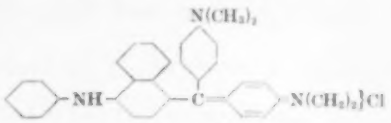
| Dye content, % ... ... | | 0·39 | 0·845 | 1·34 | 2·96 |
|---|---|---|---|---|---|
| [A⁻] value ... ... ... | | 0·0310 | 0·047 | 0·0642 | 0·120 |
| | Molality | | | | |
| KCl | 0·1 | 2·8<br>3·8 | 3·1<br>5·6 | 5·3<br>7·6 | 10·5<br>14·2 |
| | 0·05 | 6·3<br>7·6 | 7·7<br>11·0 | 10·5<br>14·8 | 20·0<br>25·3 |
| | 0·02 | 14·9<br>18·0 | 18·2<br>24·5 | 25·6<br>30·9 | 41·1<br>45·7 |
| | 0·01 | 26·4<br>31·0 | 28·3<br>39·1 | 34·7<br>46·7 | 52·8<br>62·8 |
| NaCl | 0·1 | 1·8<br>3·8 | 2·6<br>5·6 | 3·9<br>7·6 | 9·2<br>14·2 |
| | 0·05 | 4·9<br>7·6 | 6·0<br>11·0 | 7·8<br>14·8 | 17·0<br>25·3 |
| | 0·02 | 14·2<br>18·0 | 16·5<br>24·5 | 19·6<br>30·9 | 31·6<br>45·7 |
| | 0·01 | 26·4<br>31·0 | 27·1<br>39·1 | 31·3<br>46·7 | 44·6<br>62·8 |
| LiCl | 0·1 | — | — | — | 9·6<br>14·1 |
| | 0·05 | — | — | — | 17·2<br>25·3 |
| | 0·02 | — | — | — | 33·0<br>45·7 |
| | 0·01 | — | — | — | 47·5<br>62·8 |



C.I. Direct Red 2
(mol. wt. 724)

| Dye content, % | | 0·349 | 0·723 | 1·47 | 3·44 | 5·0 |
|---|---|---|---|---|---|---|
| [A⁻] value | ... | 0·0242 | 0·0329 | 0·0502 | 0·0969 | 0·1322 |
| | Molality | | | | | |
| KCl | 0·1 | 3·0<br>3·0 | 4·0<br>4·0 | 5·3<br>6·1 | 8·5<br>11·7 | 10·9<br>15·5 |
| | 0·05 | 6·0<br>5·9 | 7·2<br>8·0 | 9·5<br>12·0 | 15·9<br>21·6 | 19·7<br>27·3 |
| | 0·02 | 14·9<br>14·4 | 17·2<br>18·8 | 22·7<br>26·4 | 31·7<br>40·6 | 36·5<br>47·9 |
| | 0·01 | 24·7<br>25·5 | 28·7<br>31·9 | 33·0<br>41·4 | 44·4<br>57·3 | 48·2<br>65·0 |
| NaCl | 0·1 | 2·1<br>3·0 | 3·1<br>4·0 | 3·8<br>6·1 | 7·5<br>11·7 | 8·4<br>15·5 |
| | 0·05 | 5·6<br>5·9 | 6·3<br>8·0 | 8·1<br>12·0 | 13·5<br>21·6 | 16·4<br>27·3 |
| | 0·02 | 13·3<br>14·4 | 15·7<br>18·8 | 21·7<br>26·4 | 28·6<br>40·6 | 32·5<br>47·9 |
| | 0·01 | 23·6<br>25·5 | 26·5<br>31·9 | 31·0<br>41·4 | 41·9<br>57·3 | 47·4<br>65·0 |

TABLE III—*continued*




C.I. Basic Blue 26*
(mol. wt. 648)

| KCl, molality | | ... | 0·1 | 0·05 | 0·02 | 0·01 | [A⁻] value (calc.) |
|---|---|---|---|---|---|---|---|
| Dye | 0 | ... | 2·0 | 5·5 | 14·0 | 23·3 | 0·018 |
| | 4·1% | ... | 1·0 | 2·6 | 7·6 | 13·5 | −0·039† |

* Experimental potentials only
† Sign of charge reversed

similar, the sheet method was not used further.

It will be observed that there is fair agreement between the observed values of the Donnan potential $\psi_2$ and those calculated from a constant [A⁻] value of 0·018, for all three alkali-metal chlorides.

The discrepancy observed with the most dilute solution ($m$ = 0·001) is probably due, as pointed out by Neale and Standring [1], to the difference of about one unit between the external and the internal pH, corresponding to a Donnan potential of around 58 mv. This suppresses dissociation of the weakly acid carboxyl groups, and causes the [A⁻] value to fall.

The observed values of the Donnan potential and those calculated on the assumption of complete electrolytic dissociation of the dyes are also compared in Fig. 2 and 3.

## DISCUSSION OF RESULTS

The observed potentials of dyed cellulose increase with the dye content in the case of the two anionic dyes, and decrease with the cationic dye, as expected. The effect of the adsorbed dye upon the cellulose potential is, however, in all cases less than the theoretical. This is probably due to the occlusion of the counter-ions [7] reducing the [A⁻] value by about one-third. Such occlusion is a well established feature of the behaviour of polyelectrolytes generally.

A further source of uncertainty lies in the unknown ionic activity coefficients in the system. The results do, however, substantiate the general hypothesis of ionic adsorption, and the division of the fibre–dyebath system into two homogeneous phases, as a useful first approximation in the theory of dyeing.

DEPARTMENT OF CHEMISTRY
MANCHESTER COLLEGE OF SCIENCE AND TECHNOLOGY
MANCHESTER 1

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[5] Meyer, K. H., and Sievers, A. M., *Helv. chim. Acta*, **19**, 649 (1936).
[6] Neale, S. M., *Trans. Faraday Soc.*, **43**, 338 (1947).
[7] Vickerstaff, T., *op. cit.*, p. 84.

# Relation between Physical State and Rate of Fading of Dyes

G. BAXTER, C. H. GILES, and W. J. LEWINGTON

A theoretical analysis of curves expressing the rate of fading of dyes in different physical forms is given, illustrated by typical examples of the fading of dyes in transparent films, measured spectrophotometrically. Molecularly dispersed dye fades according to an approximate first-order law (exponential decrease of rate), and dye in large particles according to an approximate zero-order law (constant rate). In aggregated particles fading probably occurs only at the surface. First-order fading occurs with some water-insoluble dyes in "solid solution" in a hydrophobic substrate (collodion), and some surface-active dyes in a hydrophilic substrate (methylethylcellulose) appear to exist mainly as molecular dispersions, since they also give first-order fades. In contrast, entirely aggregated dyes, e.g. water-insoluble dyes prepared in substance and incorporated in hydrophilic films, show zero-order fades.

The form of the fading-rate curves of most water-soluble dyes, however, suggests that the dyes are present in both forms, i.e. as a heterogeneous mixture of single molecules and aggregated material, the proportion of each varying according to the physical properties of the dye. The heat generated during irradiation causes some of the larger aggregates to break up. This may lead to a slight initial rise in depth of colour or, in special cases, to a fading rate which *increases* with time.

The forms of the appropriate fading-rate curves show that a disaggregating agent (phenol) and a cationic surface-active dispersing agent, when mixed with acid dyes in films, decrease the amount of dye aggregate present.

The fading rates of the Procion (ICI) reactive dyes in cellulose film are of the same form as those of most water-soluble dyes, and appear to demonstrate that most of the dye is in some form of aggregated state or is enmeshed in crystalline regions of the fibre.

The results explain why the light-fastness grading of dyed fabrics may differ with the extent to which fading has progressed when the assessment is made.

The light fastness of dyes is determined by both chemical* and physical factors, especially by their physical state in the fibre [1, 2]. The present paper describes an analysis of the fading rates of a few typical dyes in several physical forms, and shows how the shape of its fading-rate curve may be used to diagnose the physical state of a dye.

## PREVIOUS WORK

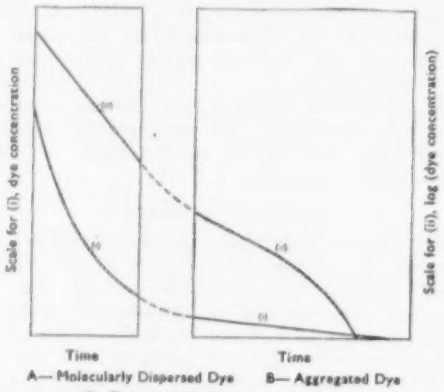
The rate of fading of dyed fabrics has been studied by Barker, Hirst, and Lambert [3], who measured the loss of dye by means of the Lovibond Tintometer, and Cunliffe and Lambert [4], who used the Guild trichromatic colorimeter. These authors found a linear relationship between the square root of the time of exposure and the amount of dye faded. Barker, Hirst, and Lambert also found that the amount of dye faded was approximately the same whatever the initial depth of colour. Presumably this would represent (as it would do in fading in films) a reaction approaching zero order, a condition which is discussed below.

## THEORETICAL FORMS OF FADING-RATE CURVES

The course which fading should take when the dye is present in any one of three different physical states, viz. (*a*) molecularly dispersed, (*b*) aggregated, or (*c*) equilibrium mixture of (*a*) and (*b*), is shown in Fig. 1, which gives hypothetical curves of rate of loss of a dye made by plotting residual dye concentration or its logarithm against time.

### *Rate decreasing with Time*

A dye at a low or moderate depth, and in the form of a true dispersion or monolayer, i.e. a state in which every molecule is equally accessible to the illumination and to the chemical species causing fading, should fade according to a first-order law; i.e. the rate should decrease exponentially with time, so that the *proportion* lost over a fixed period is the same whatever the initial concentration. The curve showing the decrease in the percentage of unchanged dye present with time is in this case a normal exponential curve (Fig. 1, iA), and when the logarithm of the dye percentage is used for the vertical axis, the curve is a straight line (Fig. 1, iiA).




(i) Dye concentration
(ii) Logarithm of dye concentration

FIG. 1— Theoretical Fading-rate Curves

### *Rate remaining constant*

It is supposed that, as a first approximation, only dye molecules which are readily accessible to the surrounding atmosphere can fade. Therefore, if the dye is present in a substrate entirely as large particles, only the molecules on exposed surfaces fade. If the particles are large, their surface area, and so the amount of dye which can be considered available for fading, remains almost constant throughout the early stages. The rate of fading is therefore almost constant, and the reaction may be said to approach zero order, or the *amount* lost over

* The present paper is not concerned with the cases in which chemical abnormalities, such as marked catalytic fading effects, determine fading rates.

any fixed time interval should be the same, however much may be present at the start. This condition gives a straight-line relationship between time and amount of dye present (Fig. 1, iB), and a line with increasing downward slope when the logarithm of the amount of dye is plotted against time (Fig. 1, iiB).

### *Rate increasing with Time*

Under some conditions, discussed below, large particles may disintegrate under the influence of heat during irradiation, and the fading rate may then actually increase with time.

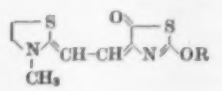
### *Complex Systems*

The conditions described above are idealised, and except in special circumstances real dye–substrate systems must be more complex and likely to contain a mixture of single molecules and aggregates of a variety of sizes. The course of fading of a simple mixture of molecules and large particles would be represented by the whole curves shown in Fig. 1 (A + B) (i and ii), the single molecules fading first, and this is likely to be the type of relationship between time and amount of unchanged dye which will occur in many cases in practice.

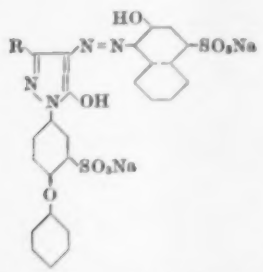
A number of actual fading-rate curves illustrative of the above theoretical principles are shown in Fig. 2–15, and their significance is described below. These are chosen more or less at random from several hundreds of fading-rate measurements which have been made in the course of recent investigations. The dyes indicated in Table I have been used to determine the present curves.

TABLE I

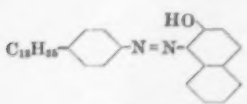
**List of Dyes investigated**



I— (*a*) R = *n*-$C_8H_{17}$; (*b*) R = *n*-$C_{14}H_{29}$



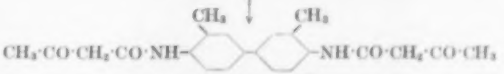
II— (*a*) R = $CH_3$; (*b*) R = *n*-$C_{17}H_{35}$



III

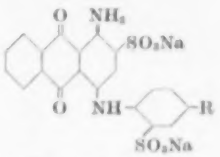
Brentamine Fast Red B Base
(C.I. Azoic Diazo Component 5)

Diazotise and couple

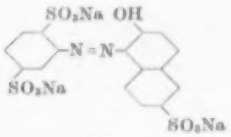


Brenthol AT
(C.I. Azoic Coupling Component 5)

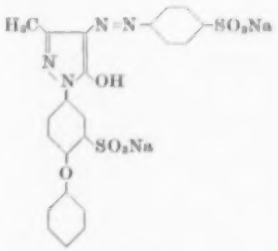
IV



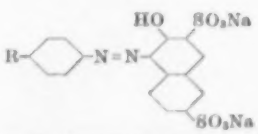
V— (*a*) R = H; (*b*) R = *n*-$C_{12}H_{25}$



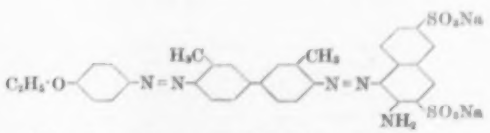
VI



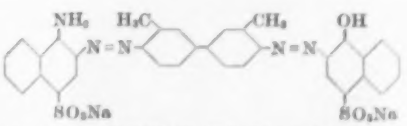
VII



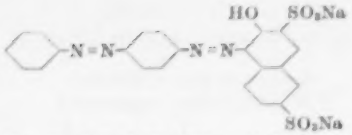
VIII— (*a*) R = H (Orange R— C.I. Acid Orange 14); (*b*) R = *n*-$C_4H_9$; (*c*) R = *n*-$C_{12}H_{25}$; (*d*) R = *n*-$C_{16}H_{33}$



IX— Congo Orange R (C.I. Direct Orange 13)



X— Congo Corinth B (C.I. Direct Violet 21)




XI— Cloth Red 2R (C.I. Acid Red 150)

### FADING RATES OF MOLECULARLY DISPERSED DYES

Two types of dye were prepared in forms believed to be molecularly dispersed, the water-insoluble *mero*cyanine dyes I(*a*) and I(*b*) in collodion, and the water-soluble surface-active azo dyes II(*a*) and II(*b*)* in methylethylcellulose. The two *mero*cyanines are believed to exist in collodion as a "solid solution" [1], and the two azo dyes as a monolayer on the internal surface of the methylethylcellulose film. All these dyes give true first-order fading curves (Fig. 2 and 3), consistent with molecular dispersion†.

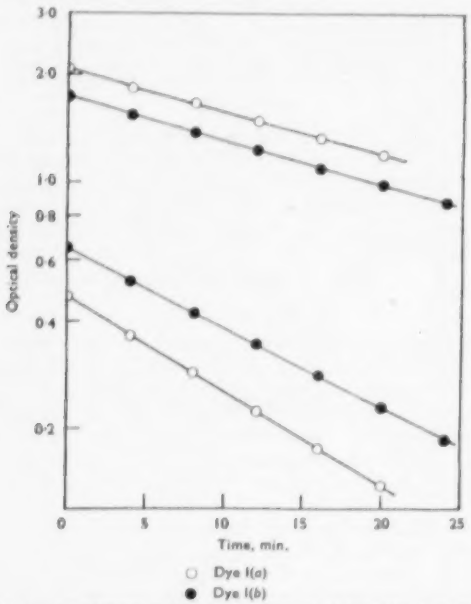



FIG. 2— Logarithmic Fading-rate Curves of Molecularly Dispersed Dyes— *mero*Cyanine Dyes in Collodion Films

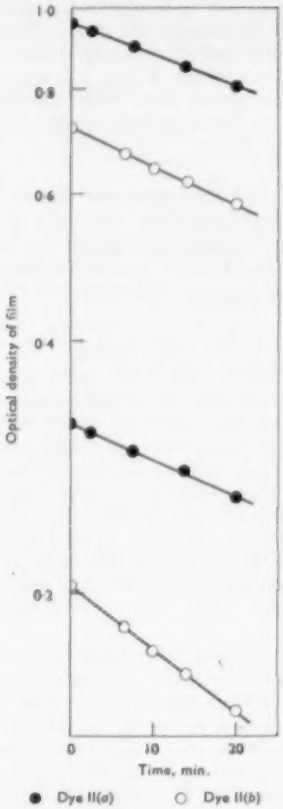



FIG. 3— Molecularly Dispersed Dyes— Acid Dyes in Methylethylcellulose (logarithmic)

### NORMAL FADING BEHAVIOUR OF PARTICULATE DYES

Three types of dyed material have given rate curves with zero-order characteristics— (*a*) a water-insoluble dye (III) precipitated from acetone into aqueous methylethylcellulose solution, which is then dried to form a film (Fig. 4); (*b*) a water-insoluble dye (IV) prepared in regenerated cellulose film by the usual process of impregnation with the coupling component followed by treatment with a diazonium salt (Fig. 5); and (*c*) cast gelatin films coloured with two water-soluble sulphonated anthraquinone acid wool dyes (blues) (Fig. 7). The dye in (*a*) obviously must consist of relatively large particles, and there seems no doubt that the dyes in (*b*) and (*c*) also are largely particulate, that in (*b*) being prepared by an insolubilisation process, and those in (*c*) being very similar in structure to the Solway Blues (C.I. Acid Blue 43 and C.I. Acid Blue 45) which Astbury and Dawson [7] found to be crystalline in wool. The crystallinity of (*c*) must account for their high fastness. The initial rise in the curves of Fig. 5 and 7 is additional evidence of the presence of particles; this is discussed below.

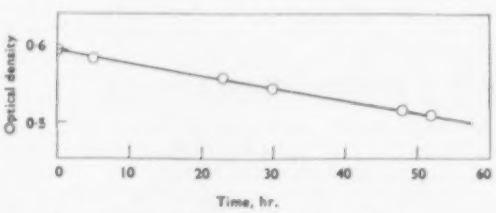



FIG. 4— Aggregated Dye— Water-insoluble Dye (III) in Methylethylcellulose

* Dye II(*b*) is surface-active on water alone, II(*a*) only in presence of a cationic agent [5].

† The curvature of the "CF" line for the dyes I(*a*) and I(*b*) [6] is now considered to be due to the "diffusion-restriction" effect operating either in the entry of oxygen and moisture or in the removal of fading decomposition products, which is believed to retard fading in hydrophobic fibres [2], rather than to the cause previously suggested [1]. This effect will become more pronounced, and hence the fading will be more retarded, as the depth of dyeing increases.

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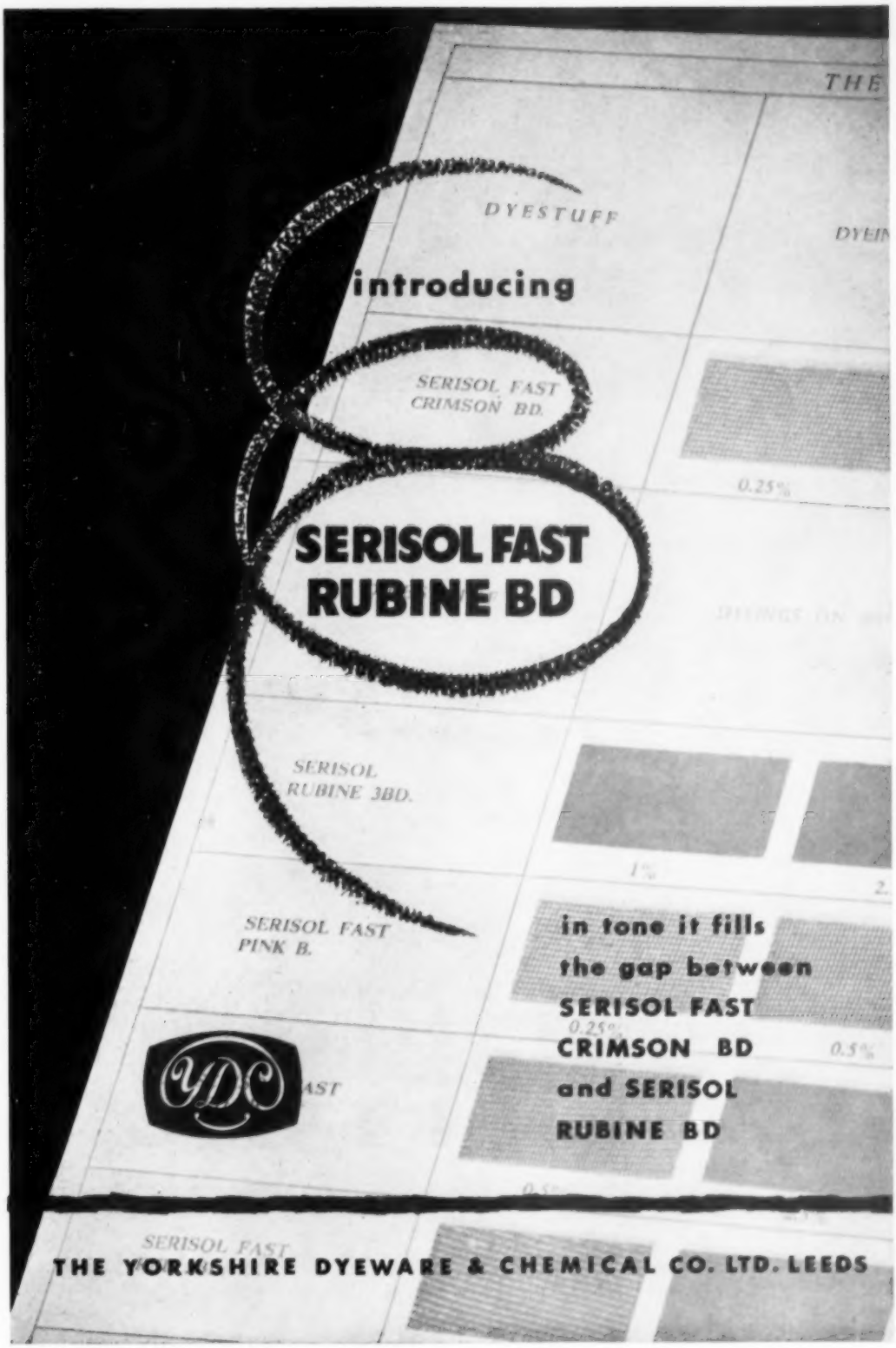
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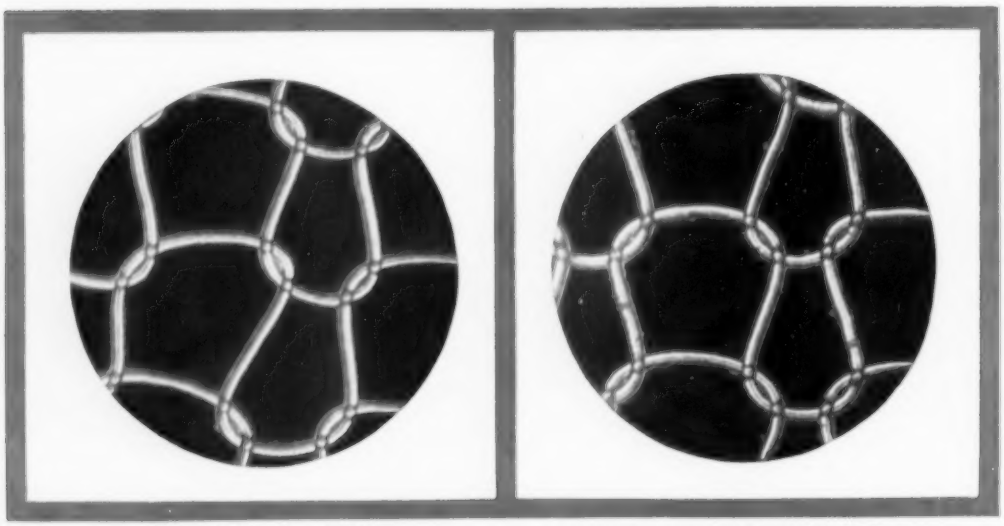

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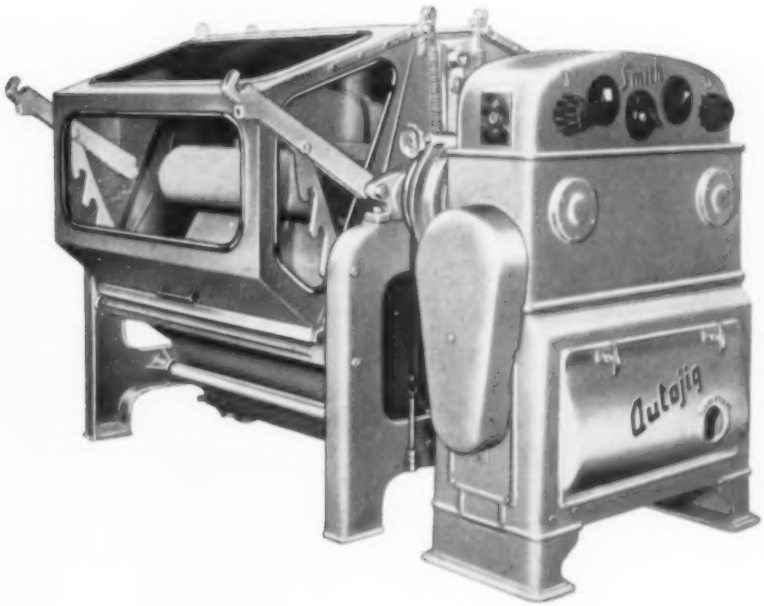


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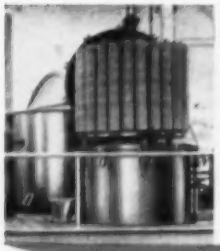

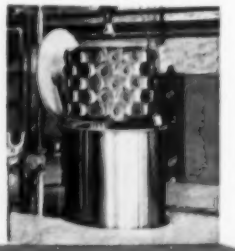

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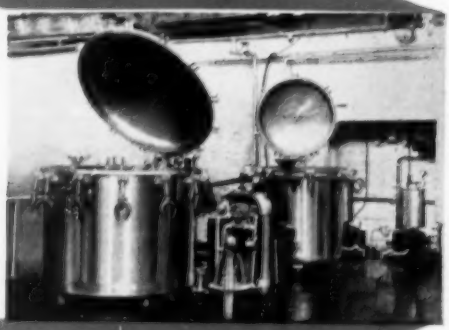

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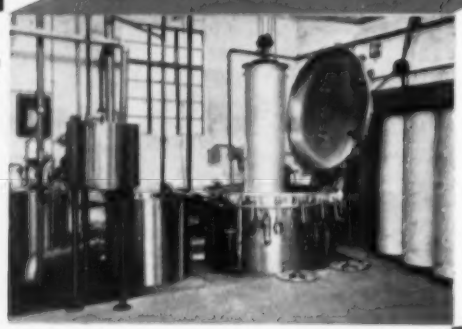

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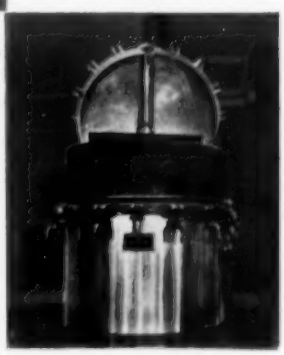

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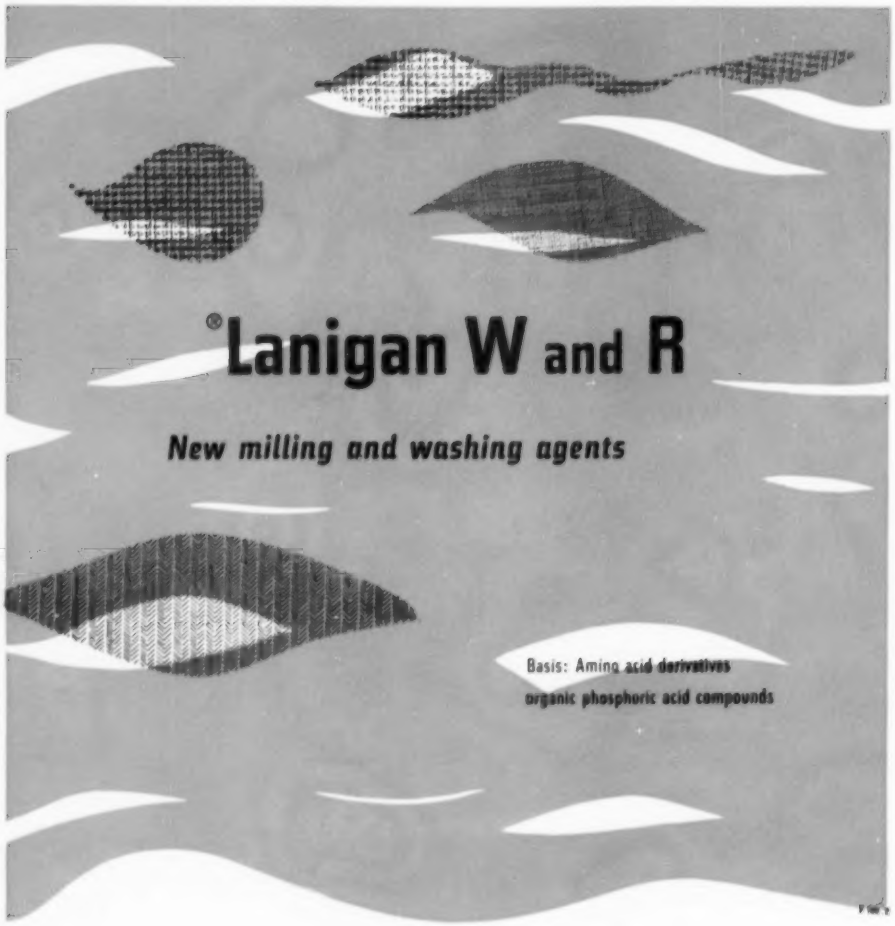


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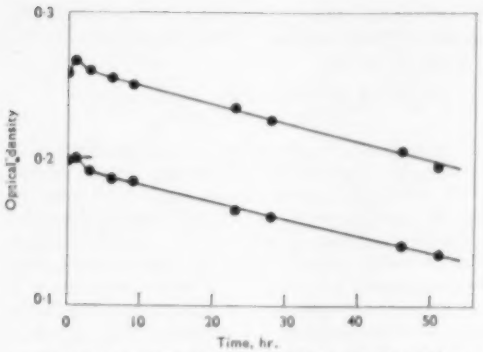



FIG. 5— Aggregated Dye— Azoic Combination (IV) dyed on Cellophane Film

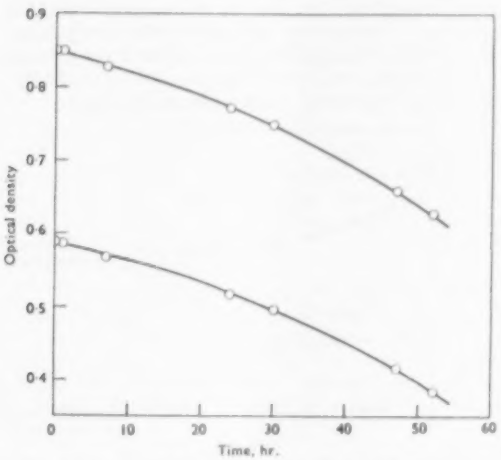



FIG. 6— Aggregated Dye— Azoic Combination (IV) dispersed in Methylethycellulose Films

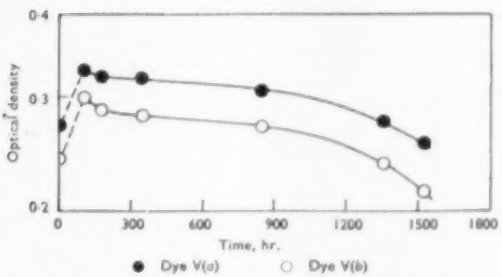
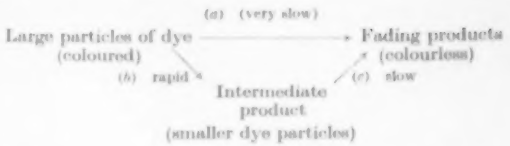



FIG. 7— Anthraquinone Acid Dyes in Gelatin Films (logarithmic)

## ABNORMAL FADING BEHAVIOUR OF PARTICULATE DYE

When the same dye as that used for colouring the films used in the tests for Fig. 5 is incorporated in methylethylcellulose films after preparation in substance, the fading-rate curves have a quite different shape (Fig. 6). The rate of decrease of optical density now *increases* with time. This can be explained by a pattern of reactions shown thus—




The original large dye particles can fade to the end-products by two alternative routes— by a direct route (*a*), which is very slow, or by a more rapid, two-stage route, in which they quickly break up into smaller particles (still coloured) (reaction *b*), under the influence of the heat of the lamp, and these small particles then fade by a slow reaction (*c*), which is, however, more rapid than (*a*). The small coloured particles, although fading more quickly than the large ones, are formed so rapidly that they become steadily more numerous. Thus, in effect the amount of dye available for fading increases with time, and so, therefore, does the reaction rate.

The optical density of a given weight of dye may increase a little as its particle size decreases, and an initial rise in a fading-rate curve (cf. Fig. 5 and 7) shows that this increase at first outweighs the decrease due to fading. An initial rise of this sort is in fact observed in a number of cases [cf. 8].

## DYES FADING AS "MIXED" FORMS

In many cases water-soluble dyes fade in a manner suggesting the presence of a mixture of single molecules and aggregated material. A typical example is illustrated by Fig. 8 (cf. Fig. 1, iA and B), in which there is an initial rapid loss of dye by an approximately first-order reaction (decreasing rate) followed by a much slower loss approaching zero order (constant rate). Some further examples of this behaviour are given in Fig. 9–15. It appears that dyes VIII(*b*)–(*d*) are more aggregated than VIII(*a*), for the initial rapid loss of dye is much greater for the first dye (Fig. 10). Similarly, in the pair of dyes with large molecules (IX and X) that with the water-solubilising groups at only one end, which is the more surface-active [cf. 5], has a more rapid initial fade (Fig. 11), consistent with a higher proportion of single molecules in a monolayer and less aggregated material present. This will be discussed more fully in a later paper.

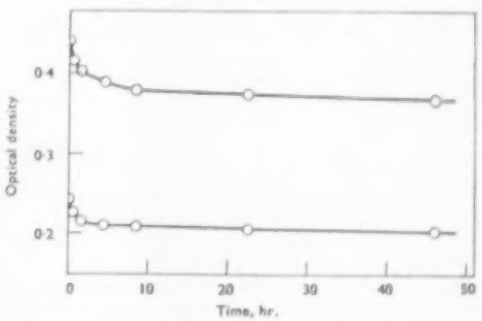



FIG. 8— Azo Acid Dye (VI) in Gelatin Films

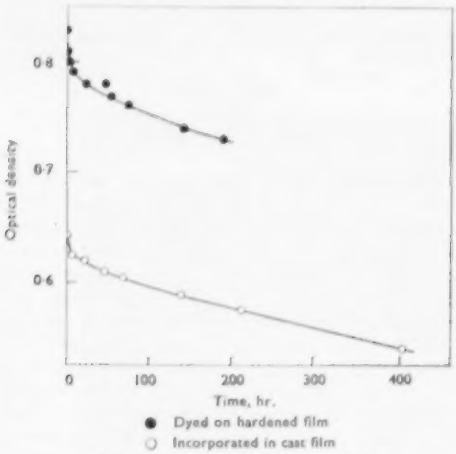



FIG. 9.—Azo Acid Dye (VII) in Gelatin Films

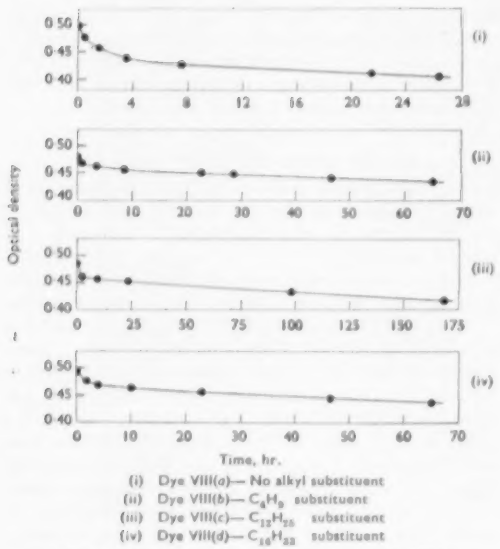



FIG. 10.—Homologous Azo Acid Dyes in Gelatin Films

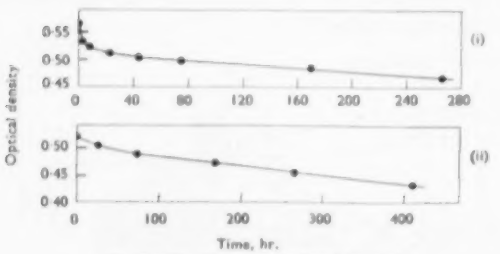



FIG. 11.—Disazo Dyes in Gelatin Films

The above results were obtained with gelatin films prepared by casting and drying mixed solutions of dye and gelatin in water, but similar results are obtained when a preformed uncoloured (hardened) gelatin film is dyed by immersion in a dyebath (Fig. 9).

### EFFECT OF DISAGGREGATING AGENTS UPON FADING RATES

If aggregates of dye are present in films, then certain hydrogen-bonding agents, e.g. phenol, which is known to cause disaggregation of some azo dyes in aqueous solution [9], should reduce their size. Accordingly, films of an azo dye (VIIIc), which, from its fading behaviour, is believed to be considerably aggregated in cast films, were prepared with and without the addition of phenol, and the fading rates were determined. The curves (Fig. 12) show a striking difference between the rates of fading of the dye in the two states: without phenol the fading curve is almost linear, and apparently the dye is almost entirely in the form of aggregates; in presence of phenol the rate curve in the early stages has the shape characteristic of molecularly disperse dyes, though in later stages it is much flatter, probably because of residual large particles.

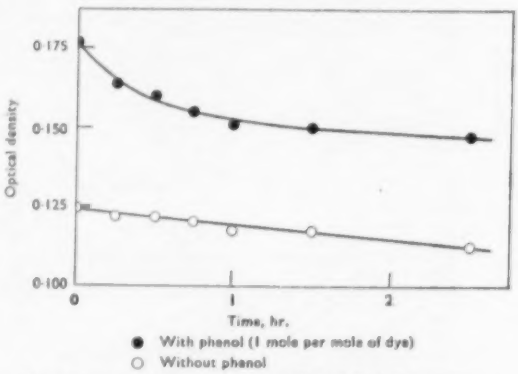



FIG. 12.—Azo Acid Dye (VIIIb) in Methylethylcellulose Films, illustrating Disaggregating Effect of Phenol

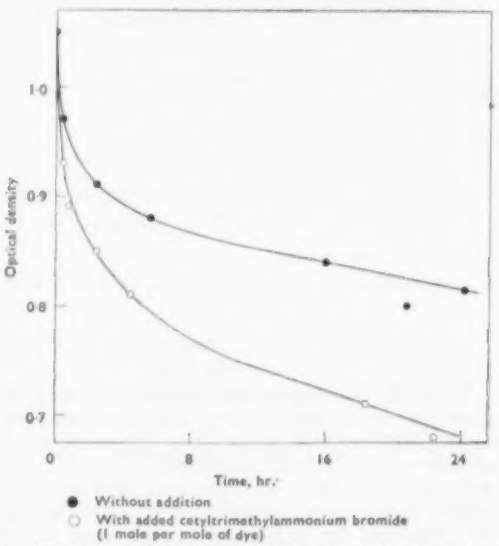



FIG. 13.—Disazo Acid Dye (XI) in Gelatin Films, illustrating Effect of Surface-active Dispersing Agent

A very similar change in rates occurs when a cationic surface-active agent (cetyltrimethylammonium bromide) is mixed with a sulphonated acid dye in gelatin film (Fig. 13). This agent is known to facilitate the formation of a monolayer of some acid dyes on water [5], and it presumably does the same in the gelatin film. Another cationic surface-active agent was shown previously [1] to decrease the fastness of direct cotton dyes (in Cellophane film), an effect well known to occur in practice when this type of agent is used to aftertreat such dyes on cellulosic fibres *cf.* [2].

## REACTIVE DYES

These new dyes are said to react directly with the fibre, forming stable covalent bonds [10], and thus to differ from all other types of dye, which are adsorbed by reversible physical forces. The mode of combination of the Procion (ICI) dyes might seem to imply that they must exist only as single molecules in the fibre. If so, they would be expected to fade very rapidly, but in fact they have high fastness to light. It has been suggested that this high fastness could be explained in two ways— either (*a*) because of their much greater than normal chemical reactivity with fibre molecules they can break fibre–fibre bonds in crystalline regions and so penetrate where normal dyes do not, being then less accessible to air and moisture; or (*b*) because the fibre moelcules in amorphous regions have a certain freedom of movement in water and can be considered solvated, they may form mixed dye–fibre aggregates. Whatever may actually occur, it seems clear that these dyes cannot be in normal molecular dispersion, as they have such very good light fastness.

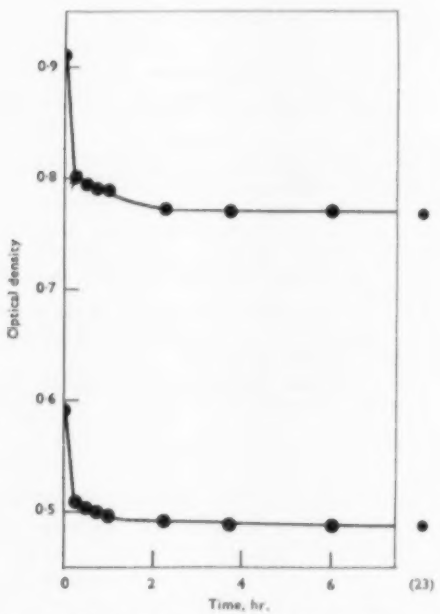



FIG. 14— Procion Yellow RS (ICI) on Cellophane Film

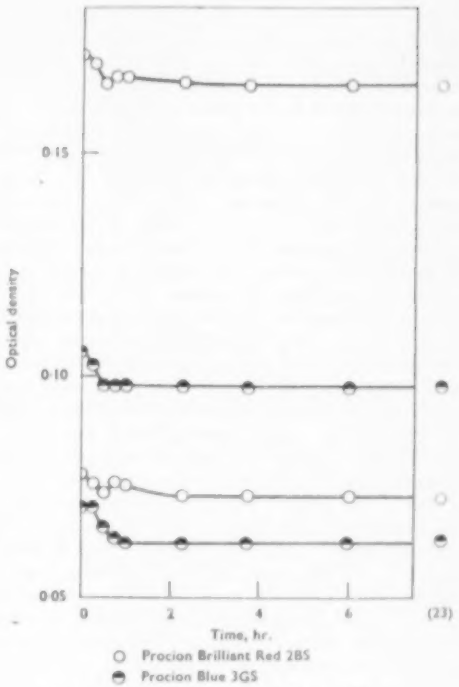



FIG. 15— Procion (ICI) Dyes on Cellophane Film

Rate measurements have now been made on Cellophane regenerated cellulose sheet dyed with each of the first three Procion dyes. The rate curves (Fig. 14 and 15) appear to confirm that, except for a very small proportion which has an initial rapid first-order fade, the dyes are not present in normal molecular dispersion. They fade very slowly and at a more or less constant rate, resembling in fact more ordinary dyes.

## CONCLUSIONS

Dyes present in a substrate entirely in molecularly dispersed or monolayer form fade according to an approximately first-order law, and those present entirely as discrete particles fade according to an approximately zero-order law. Most ordinary water-soluble dyes, however, appear to consist partly of monolayer material, which is responsible for an initial rapid fade, and partly of aggregated dye, which causes the subsequent rate to remain nearly constant.

Aggregates of dye at first tend to break up in the heat of the illumination, and this process can in some cases cause the fading to accelerate as exposure continues.

Disaggregating or spreading agents added to dyed films alter the shape of the fading-rate curves in a manner which shows that they reduce the proportion of aggregated dye present.

The results of this investigation may be taken as corroborative evidence that light fading of aggregated dye takes place mainly at the surface of the aggregates.

### TECHNICAL CONSIDERATIONS

The results described in this paper explain why some dyes are given the same light-fastness grading when judged after a considerable degree of fading as when judged in the earliest stages, while others may apparently either improve or deteriorate in fastness as the time of exposure is increased. Thus Morton [11] quotes fastness gradings given to a variety of dyes after increasing degrees of fading: three direct dyes on (crease-resisted) viscose rayon improve in grading with increase in exposure (in a Fade-Ometer); seven vat dyes on cotton do the same (in daylight exposures); five out of eight azoic combinations on viscose rayon decrease in fastness, some very severely, on prolonged daylight exposure. This last result is particularly interesting, as it apparently shows a similar behaviour to that of the azoic combination illustrated here in Fig. 6. It would thus appear particularly important to specify the extent of fade at which grading is assessed for this class of dye.

The same author also gives fading-rate curves for the *B.S.* 1006 light-fastness standards over a wide range of exposure times. The method of plotting differs from that used here, and the curves shown are on a small scale, so that comparison with the present results is difficult, but it does appear that fading in the later stages approximates to a zero-order reaction.

### EXPERIMENTAL

The experimental technique was identical with that described in detail in earlier papers [1, 8]. The dyes either were obtained in an already purified form or were prepared by normal processes in the laboratory and purified by salting-out and recrystallisation from ethanol–water mixtures.

The dyed substrates were prepared as follows—

(i) COLLODION FILMS— The *mero*cyanine dyes and Necol (ICI) varnish were dissolved together in acetone before casting.

(ii) METHYLETHYLCELLULOSE FILMS— Cellofas A (ICI) solutions, prepared as already described [8], were cast after being mixed either with aqueous solutions of the sulphonated dyes II(*a*) and II(*b*) (Fig. 3), with an acetone solution of a water-insoluble dye (III) (Fig. 4), which was thereby precipitated, or with an aqueous dispersion of the insoluble dye IV, prepared in substance (Fig. 6).

(iii) CELLOPHANE REGENERATED CELLULOSE FILM was dyed with dye IV by the normal process of impregnation with the coupling component and subsequent coupling. The dyed film was well rinsed but not soaped. This film was dyed with the Procion dyes by the process for cellulosic fibres described by the manufacturers [12].

(iv) GELATIN FILMS were cast as already described [8] from solutions of pure photographic gelatin mixed with the appropriate dye solutions.

(v) HARDENED GELATIN was prepared by casting an uncoloured gelatin film and treating in a cold 4% (wt./vol.) aqueous solution of formaldehyde for 15 min., the film then being rinsed well and dyed at 60°c. in an aqueous solution of the dye VII without other addition for 30 min.

An Osira (G.E.C.) 400-w. mercury-vapour lamp was used for irradiation, measurements of dye content being made, at intervals during fading, by means of a Unicam SP 500 photoelectric spectrophotometer. Dyed films compared here against each other were irradiated together.

* * *

The authors thank Professor P. D. Ritchie for his encouragement and interest; the following for gifts of materials: Dr. D. M. Stead and James Anderson & Co. (Colours) Ltd.; Mr. G. H. Dowse and Courtaulds Ltd.; Dr. E. B. Knott and Kodak Ltd.; Mr. R. B. Collins, Mr. D. J. Fry, and Ilford Ltd.; Mrs. S. M. Neal, Dr. M. A. T. Rogers, and Imperial Chemical Industries Ltd. (Dyestuffs Division); Imperial Chemical Industries Ltd. (Nobel Division and Paints Division); and Mr. D. S. E. Campbell and Mrs. R. Catchpole for making the experiments the results of which are shown in Fig. 13–15 respectively. They also thank the Wool Textile Research Council for a financial grant for materials and a scholarship to one of them (G.B.).

COLOUR CHEMISTRY RESEARCH LABORATORY
DEPARTMENT OF TECHNICAL CHEMISTRY
ROYAL COLLEGE OF SCIENCE AND TECHNOLOGY
GLASGOW C.1

(*Received 19th December 1956*)

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[1] Baxter, Giles, McKee, and Macaulay, J.S.D.C., **71**, 218 (1955).
[2] Giles, *ibid.*, **73**, 127 (April 1957).
[3] Barker, Hirst, and Lambert, *ibid.*, **43**, 263 (1927).
[4] Cunliffe and Lambert, *ibid.*, **48**, 59 (1932).
[5] Cameron and Giles, *J.C.S.*, 3140 (July 1957).
[6] Baxter *et al.*, *loc. cit.*[1], Fig. 5.
[7] Astbury and Dawson, J.S.D.C., **54**, 6 (1938).
[8] Chipalkatti, Desai, Giles, and Macaulay, *ibid.*, **70**, 487 (1954).
[9] Alexander and Stacey, *Proc. Roy. Soc.*, **A 212**, 274 (1952).
[10] Vickerstaff, T., J.S.D.C., **73**, 237 (June 1957).
[11] Morton, *ibid.*, **65**, 597 (1949).
[12] Imperial Chemical Industries Ltd. Dyestuffs Division, *Technical Information* (*Dyehouse*) No. 305 (1956).

## ERRATA

**The Light Fastness of Dyed Fibres—A Statistical Study**
(C. H. Giles, J.S.D.C., **73**, 127–160 (April 1957))

On p. 139 Table IV should include the reference [379]. In Fig. 12 the fibre descriptions of the first three pairs should be reversed. In the caption to Fig. 13 the reference should be to *private communication from Mr. R. J. L. Skelton and the United Turkey Red Co. Ltd.*, and not as given.

On p. 147 in footnote ‡ to Table VIII the word "cotton" in the third line should be *viscose rayon*.

On p. 149 in the last paragraph of the first column (sixth line) the phrase "(without carrier)" should read *(data for heavy depths given for fibre dyed with Tumescal D (ICI) as carrier, but this does not affect fastness—cf. Table IV above)*.

**Testing the Physical Properties of Sizing Liquids**
Gebrüder Sucker (J.S.D.C., **73**, 364 (July 1957))—The number of the patent should be *BP* **768,472.**

# Notes

### Meetings of Council and Committees

**July**

Council — 3rd

Publications— 16th

*Colour Index* Editorial Panel— 25th

Examinations Board— 18th and 19th

Diplomas— 3rd and 31st

Finance and General Purposes— 3rd

Terms and Definitions— 26th

Symposium— 17th

Textbooks and Monographs Subcommittee— 16th.

### Deaths

We regret to report the loss by death of Mr. A. Anderson, Mr. P. G. Jones, Mr. C. Leach, Mr. W. N. Lepine, and Mr. E. Payne.

### Election of Fellows

Christopher Barrow
(*Manchester; Chief Chemist and Colourist, English Sewing Cotton Co. Ltd.*)

John Coyne
(*Devonport, Tasmania; Dyeing and Finishing Manager, Tootal Broadhurst Lee Co. Ltd.*)

Rhys Jenkin Loveluck
(*Grangemouth, Scotland; Works Manager's Department, Imperial Chemical Industries Ltd. Dyestuffs Division*)

Herbert Wilson Partridge
(*Ambergate, Derbyshire; Chief Chemist, Stevensons (Dyers) Ltd.*)

Jack Augustus Radley
(*Reading; Research and Development Consultant*)

Charles Dennis Tinsdeall
(*Madras, India; Chief Chemist, Buckingham & Carnatic Co. Ltd.*)

Percival J. Wood
(*Carlton Hill, N.J., U.S.A.; Technical Director, Royce Chemical Co.*)

### Notes for a Laboratory Course in Dyeing

Notes on laboratory experiments suitable for a course in practical dyeing have been compiled by Dr. C. H. Giles, Senior Lecturer in Colour Chemistry at the Royal College of Science and Technology, Glasgow. The Society expects to publish them under the above title during September 1957, so that they will be available to students in time for the beginning of the new academic year. The price will be 17s. 6d., and orders may be sent direct to the Society.

### AATCC Awards

Dr. H. C. Chapin and Mr. A. E. Sampson, who recently retired as national secretary and treasurer respectively of the American Association of Textile Chemists and Colorists, have been elected Honorary Members of the Association. The only other Honorary Members are now Mr. W. H. Cady and Mr. W. R. Moorhouse, and only three other such awards have been made in the history of the Association.

The Olney Medal is to be awarded to Mr. P. J. Wood, F.S.D.C., who received his technical training at the Yorkshire College (now the University of Leeds), served his apprenticeship with William Grandage & Co. in Bradford, and returned to the University as Lecturer in the Department of Colour Chemistry and Dyeing. Mr. Wood was one of the earliest Junior Members of the Society, and has served the Association as president and on many of its committees.

### S.V.C.C. Meetings

The Schweizerischer Verein der Chemiker-Coloristen has arranged for 27th and 28th September 1957 visits to the Schaeffer mills in Pfastatt, the potassium mines in the neighbourhood, the Laederach–Dungler bleachworks, and the Textile Institute in Mulhouse. On 2nd and 3rd November 1957 there will be a social gathering on the Rigi near the Lake of Lucerne. On 7th December 1957 a meeting with lectures will be held in Winterthur. The S.V.C.C. has issued a warm invitation to members of the Society to attend these functions.

# New Books and Publications

## Tafeln zur Prüfung des Farbensinnes
### (21, neubearbeitete Auflage der Stilling–Hertel'schen Tafeln)

Edited by Karl Velhagen Jr. Pp. 15 + 19 (37 colour plates). Stuttgart: Georg Thieme Verlag 1952. Price, DM 18.40.

Among the well tried and best known methods of testing colour vision is the series of diagrams based on apparent colour uniformity (pseudo-isochromasy) devised by the German anatomist, B. Stilling. E. Hertel developed a series of tests largely based on Stilling's principles which formed the 20th edition of the Stilling–Hertel Plates.

The present (21st) edition also contains some pseudo-isochromatic plates, although these are modified and revised compared with those in the previous edition. Thus in Plates 3–10, 12–18, 23, and 24 a person of normal colour vision readily picks out a number or a letter against a background of spots usually coloured in a contrasting hue. Red-green dichromats, on the other hand, can scarcely read the script.

There are also some pseudo-non-isochromatic plates (No. 11, 19, 20, 21, 22, and W 1), aptly described as *Vexier* (puzzle) plates. In these a second bright colour is brought against the contrastingly coloured pseudo-isochromatic writing, and the red-green-blind person sees an entirely different number or figure from the normal person.

The above tests, however, do not claim to be able to distinguish protanopes (red-blind) from deuteranopes (green-blind), or anopes from anomalous trichromats, who have colour weakness. Special attention is paid to tritanopia (blue-blindness), which is indicated by incorrect reading of Plates 21–24.

A further feature of the tests is the importance attached to colour contrast and colour selection, which are claimed to be useful in doubtful cases and in milder forms of colour weakness. Plates K 1–K 7 are constructed quite differently from the preceding diagrams, and the colour-weak person finds difficulty in judging the correct colour of a central (grey) spot placed on a background of red or green. On a green ground they declare the grey spot to be red, and on a red ground green; i.e. they observe an apparently increased colour contrast which in practice might have serious consequences, as in misjudging the colour of traffic signals.

The remaining four plates, F 1–F 4, embody the exactly opposite principle of diminishing colour contrast ("*Florcontrast*"). Grey–black letters or figures are viewed on a saturated coloured ground through tracing paper, which disguises them and renders them ill defined. To a normal person the grey colour of the script changes over to the complementary colour to the background, thus reversing the effect of the plates of increasing colour contrast (K 1–K 7). Anomalous trichomats, who have a lower sensitivity to the ground colours, find difficulty in recognising the shape of the writing through the tracing paper.

The detailed instructions for carrying out the tests are given with typical German thoroughness. General procedure is followed by selection tests in doubtful cases and by special questions for specific purposes, e.g. detection of trianopia.

Short cuts and simplification of the testing procedure are given; cases are detailed where examination by a medical eye specialist is considered necessary: also hints on the possibilities of control and on the recording of results. Simulators (pretenders) and dissimulators (concealers) of defective colour vision are faced with a formidable series of tests and questions designed to trap them.

Professor Velhagen has compiled a fascinating series of plates, and many persons seriously interested in testing colour blindness will be anxious to try out the various principles embodied in the tests. A few preliminary trials suggest that some of the plates are extremely sensitive to colour weakness, but only a prolonged and extensive series of tests can confirm the claims made for them. An English translation would be most welcome, for there is much useful information concerning colour blindness in the general explanation which precedes the technical instructions.

Use of the test would be much simplified if a concise key were included, giving the actual figure(s) or number(s) which a "colour-efficient or a "colour-inefficient" person is expected to read. In some *Vexier* plates it is not stated what a normal person should see, e.g. No. 19, 21, 22, and W 1. Plate W 2 looks interesting and is divided into two equal rectangles, but nowhere in the booklet of instructions can be found any reference to what it signifies.

F. Jordinson

## International Discussion of Problems in Colour Metrics

Edited by Manfred Richter. *Die Farbe*, **4**, 113–328 (Dec. 1955). Göttingen: Musterschmidt-Verlag. Price, DM 23.40.

The discussion was concerned essentially with three important fields of work and is reported in the three sections—(1) *Problems of Chromatic Adaptation and of the Influence of Surrounding Fields*, (2) *Problems of Colour Vision, especially of Tritanopia and of Viewing Very Small Fields*, and (3) *Problems of Colour Space, Colour Systems, and Colour Tolerances*.

The contents of the papers presented are in general clearly recorded, and it is a pity to find quite a number of typographical errors. A list of German technical colour-vision terms with English and French equivalents could, perhaps, have been included in the publication with advantage. It is unfortunate that the subject-matter should be

printed on glossy paper, which makes reading very difficult, particularly in artificial light.

In Section (1) of the *Discussion* a good deal of the work will no doubt be of interest to all those concerned with colour (particularly colour matching). In all papers, related sections could with advantage have been discussed together and results compared, particularly in those cases where divergent opinions of authors are apparent. This is more strikingly noticeable in the section on *Problems of Colour Vision* where a standardisation of the methods of investigating tritanopia and tritanomaly has been suggested.

In the main, each paper is accompanied by an adequate bibliography. The *Discussion* suffices to give a picture of the work in progress, and should stimulate further effort in difficult fields of work.

E. COATES

### Die wissenschaftlichen Grundlagen der Trocknungstechnik

By O. Krischer. Pp. xx + 400. Berlin: Springer-Verlag 1956. Price, DM 46.50.

### Experimentelle und theoretische Grundlagen der Trocknung

By A. W. Lykow. Pp. 479. Berlin: VEB Verlag Technik 1955. Price, DM 28.80.

### Neuere Erkentnisse über das Trocknen von Textilien

**Prom. Nr. 2459**

By Waclaw Ziemba. Pp. 67. St. Gallen: Zollikofer & Co. AG. 1955. Price, 9 Swiss francs.

The book by Krischer is the first volume of a larger work appearing under the title *Trocknungstechnik*. The second volume, dealing with drying equipment and methods of drying, is to be written by K. Kröll and will be published shortly. The first volume can conveniently be divided into two parts: in the first part the author deals with the physical laws governing changes of state, fluid motion, diffusion, and heat transfer; in the second part drying is presented as a phenomenon resulting from interaction of these basic processes. The treatment of heat transfer, diffusion, and fluid motion is fundamental and fairly advanced, particularly with respect to aspects which are of great importance in drying, such as fluid motion in porous substances.

The book by Lykow is a translation of the standard Russian work on the subject published in 1950 under the title *Teoriya sushki* ("Theory of Drying"). It starts with the thermodynamics of moist air, followed by a review of work done on evaporation of liquids from free surfaces. Extensive treatment is given to types of moisture binding (statics of drying) and the fundamentals of moisture movement. This is followed by a discussion of mass and heat transfer in dried substances and a detailed description of experimental techniques in drying. The mechanisms of drying are then discussed together with various accompanying phenomena, such as shrinkage and cracking. The analysis of drying curves (kinetics of drying) and a discussion of optimum drying conditions follow, and the book ends with two special chapters on infrared drying and sublimation drying.

Both authors are well known specialists in the field of drying and their books are intended to cover similar fields. Nevertheless, they can be regarded as complementary rather than alternative choices. Lykow's book is based mainly on work done in the Soviet Union with hardly any reference to Western achievements, and similarly Krischer does not include any work performed outside Germany after 1939. This clearly indicates the need for a monograph on drying in English which would combine the work reported in the above books with the achievements in this country and in the U.S.A.

In general, the advanced reader may prefer Krischer to Lykow. The former's exposition is clearer, and the use of small print for more complex relations or the introduction of numerical examples makes this even more apparent. However, Krischer makes no attempt at the classification of various materials from the point of view of their behaviour during drying, except for separate treatment of hygroscopic substances. On the other hand, Lykow differentiates between porous substances, colloidal substances, and porous colloids, and this division is adhered to throughout the book. It is also surprising that Krischer does not cover shrinkage and other mechanical deformations during drying.

Neither of the two authors mention the drying of textiles, a gap which is partly filled by Ziemba. The book is a thesis for the degree of Doctor of Technical Sciences presented at the Federal Polytechnic in Zürich. The investigations were carried out using wool and raw cotton in a tunnel drier, and the results obtained indicate that the mechanism of drying postulated by M. J. Coplan (*Text. Research J.*, **23**, 897 (1952)) hold for the drying of textiles. Experimental details and results are given together with a review of attempts to determine the apparent heat and mass transfer coefficients for this type of drying.

H. SAWISTOWSKI
D. M. NEWITT

### Polyesters and their Applications

By Bjorksten Research Laboratories Inc. Pp. viii + 618. New York: Reinhold Publishing Corporation 1956. Price, 80s. 0d.

This book, which is dedicated to the staff of the Classification Group of the U.S. Patent Office, is essentially a survey of the patent literature and other published sources of information. It is the first comprehensive account of the manufacture and applications of the polyesters and, in making this survey, the authors have performed a valuable service.

The book is prefaced by a short chapter dealing with the fundamental concepts of high-polymer chemistry, which is necessarily incomplete and, perhaps, rather superfluous in a book of this type. The manufacture of unsaturated polyesters is dealt with very fully in chapters covering raw materials, catalysts and inhibitors, fillers and

reinforcements, moulding and laminating techniques. Commercially available resins are also detailed together with some information on physical properties obtained from manufacturers' technical literature. The most important applications of resins of the unsaturated type are enumerated. The section on saturated polyesters deals with the linear fibre-forming group, which includes Dacron and Terylene, and also the di*iso*cyanate-modified polyesters.

The book is necessarily condensed, and by its very nature does not always present a complete or fully accurate account of current technology. This is well illustrated by the chapter on fibre-forming polyesters. It is, however, a valuable book of reference for experienced workers in the polyester field, and the authors are to be congratulated on performing an arduous but worthwhile task. The book is clearly set out and well indexed, and contains over 3,300 references to patent, journal, book, and manufacturers' literature.

P. T. STANDRING

## Fusion Methods in Chemical Microscopy

By Walter C. McCrone, Jr. Pp. vii + 307. New York and London: Interscience Publishers 1957. Price, $6.75.

A number of books are now being published, particularly in the U.S.A., each of which describes with a wealth of experimental detail the use of a specific modern technique in chemistry. Such books are of considerable value to the research worker, firstly as stimulants, leading him to attack old problems from a new angle, and secondly as time savers, as they fully describe methods which would otherwise have to be sought in original papers.

This volume is particularly interesting in that it shows the versatility of the microscope in organic and physical chemistry. The author defines fusion methods for the purpose of the text as including "methods and procedures which involve heating a compound or mixture on a microscopic slide". It is surprising to learn what a mass of valuable data, besides the melting point, can be obtained from this apparently simple operation.

Following the introduction, the author devotes a short chapter to describing the types of accessories, such as hot and cold stages, etc., which are required for fusion techniques.

The third chapter, under the heading *General Techniques*, forms nearly half the book. This is a thorough account of how various measurements can be made. Starting with the methods of calibrating the hot and cold stages with temperature, it goes on to describe how fusible materials can be identified by observations during melting, on the melt, and on cooling. These sections are full of interesting details on such topics as measurement of critical temperatures, polymorphic transformations, mixed fusions, etc. Methods of purification of small quantities of material are fully described and also various schemes for analysing mixtures, both qualitatively and semi-quantitatively, whilst the study of phase diagrams for binary and ternary mixtures will interest the physical chemist. Unfortunately, owing to the wealth of methods, the chapter tends to be ponderous, and what is obviously a fascinating set of techniques tends to read like a catalogue.

Chapter IV, which shows the applications of fusion methods in research, is short, as most of these applications have been discussed under the various techniques. Here the author has picked out a few research problems which have been solved by fusion methods, a particularly topical one being the study of "viscostatic" lubricating oils.

The last chapter consists of a series of tables giving melting points, temperatures at which the melt has the same refractive index as glass standards, etc. The compounds are cross-indexed by code numbers, but the method of using the latter seems rather cumbersome.

The book is well produced, the colour plates and other photomicrographs are exceptionally clear, and the author is obviously an authority on this technique. Unfortunately, the reader cannot help feeling that the cost of the specialised apparatus required tends to outweigh the results obtained, and hence that the book is needed only on the shelves of specialists working on the identification and estimation of small quantities of known organic compounds.

R. S. ASQUITH

## The Chemistry and Technology of Leather Volume I— Preparation for Tannage

Edited by Fred O'Flaherty, William T. Roddy and Robert M. Lollar. *American Chemical Society Monograph Series* No. 134. Pp. xiii + 495. New York: Reinhold Publishing Corpn. 1956. Price, 112s. 0d.

This is the first of four volumes; the subsequent ones will deal with all types of tannage, with process control of leather quality, and with the other subjects concerned with leather properties.

The previous monograph, *Chemistry of Leather Manufacture* by G. D. McLaughlin and E. R. Theis, had been published in 1945, and it became necessary to bring it up to date. The purpose of the present work is to supplement, rather than to replace, the earlier, which is therefore referred to frequently.

The histology of animal skins (W. T. Roddy 37 pp.) is covered superficially, but there follows a useful chapter on non-protein constituents of skin (R. M. Koppenhoefer, 24 pp.). The account of the chemical structure and macromolecular organisation of the skin proteins (J. H. Highberger, 129 pp.) is excellent and is a statement of the present position valuable not only to leather chemists, but to those interested in proteins in general. Although illustrated by ten photographs of works processing, the treatment of the preservation of hides and skins (F. L. De Beukelaer, 25 pp.) does not reach the technological standard to be expected. Washing and soaking (A. W. Goetz, 10 pp., no references) and the practice of unhairing (W. Morris, 28 pp.) are dealt with too briefly, but a good account of the mechanism of unhairing is given by H. B. Merrill (40 pp.). There follow chapters on the chemistry of liming (R. M. Lollar,

26 pp.), the practice of bating (H. R. Wilson, 24 pp.), and bating (J. Pfannmuller, 40 pp.). The interesting discussion of the acid- and base-binding properties of proteins (R. Saxon and M. Harris, 42 pp.) is based perforce largely on work done with wool. As few publications are available on pickling (R. Stubbings, 26 pp.), the results of a questionnaire submitted to all the major U.S. tanneries are presented, giving a good idea of current American practice and setting an example to other authors. The final chapter of the first volume concerns skin degreasing (K. G. A. Pankhurst, 27 pp.). It is excellent, concise, and balances nicely science and technology.

The book will be an essential tool for all leather chemists, but workers in related fields will also find it useful, especially because of the wealth of references. It is always difficult to edit contributions by specialists, but a tighter rein is called for. Overlap could be reduced, nomenclature standardised, and treatment made more uniform. The production is good, but expensive.

H. E. Nursten

## Symposium on Steam Quality

**A.S.T.M. Special Technical Publication 192**

Sponsored by A.S.T.M. Joint Research Committee on Boiler Feedwater Studies. Pp. v + 49. Philadelphia: American Society for Testing Materials 1957. Price, $1.75.

This publication is a reprint of the papers and discussion at the Symposium on Steam Quality presented at the Annual Meeting of the A.S.T.M. in 1956. The aim of the symposium was to review current practice in the measurement of the purity of steam from modern steam generators for power purposes.

A previous symposium presented in 1941 with the same title covered the development of the practice of steam purity measurement by determination of the conductivity of the condensate. This method was at that time beginning to supplant the measurement of steam quality in terms of its moisture content, the latter being determined calorimetrically; but the inferential method was proving insufficiently sensitive and also insufficiently informative for conditions in up-to-date steam-electric generating stations. The main reason for this was the tendency to increased boiler pressures.

The present volume thus mainly concerns itself with the purity of steam from boilers with pressures of 450 lb./sq.in. and above, up to critical and supercritical pressures. The principal problem concerns the vaporisation and mechanical carry-over of dissolved solids.

The symposium will undoubtedly be of considerable interest to power engineers, particularly in view of the mathematical presentation favoured, the full reporting of experimental details and data, and the inclusion of both the verbal and the written discussion. It is, however, only fair to warn the process engineer and technologist that he may be disappointed. The restriction of the terms of reference to high-pressure steam, logical as it is, may tend to detract from the value of the book to him.

I. R. McDougall

### New Books received

*Titration in Non-aqueous Solvents.* A. H. Beckett and E. H. Tisley. Poole: British Drug Houses Ltd. 2nd edition [1957]. Pp. iv + 43.

*1957 Yearbook.* London: British Standards Institution. Pp. iii + 484. 15s. 0d.

*Technique of Organic Chemistry.* Volume 10. *Fundamentals of Chromatography.* H. G. Cassidy. New York: Interscience Publishers Inc. 1957. Pp. xvii + 447. $9.75.

*1955–1956 Annual Report.* London: City and Guilds of London Institute. Pp. v + 104.

*The Art of Lecturing.* G. K. Clark. Cambridge: W. Heffer & Sons Ltd. [1957]. Pp. iii + 20. 1s. 6d.

*Documentazione Tessile, scientifica, tecnica e bibliografica.* Volume 3. Milan: Consiglio Nazionale delle Ricerche-Centro di Studio per l'Industria Tessile. [1956–57]. 3,000 lire.

*Neueste Fortschritte und Verfahren in der Chemischen Technologie der Textilfasern.* Zweiter Teil. *Neue Verfahren in der Technik der Chemischen Veredlung der Textilfasern. Hilfsmittel in der Textilindustrie.* Band 3. L. Diserens. Basle: Birkhäuser Verlag. 1957. Pp. xii + 933. DM 136.00.

*pH Measurements: Their Theory and Practice.* V. Gold. London: Methuen & Co. Ltd. 1956. Pp. 125. 9s. 6d.

*Ultraviolet and Visible Absorption Spectra. Index for 1930–1954.* H. M. Hershenson. New York: Academic Press Inc. 1956. Pp. xviii + 205. $12.00.

*Index of Technical Articles. No. 2.* London: Iota Services Ltd. March 1957. Pp. x + 90. £6 6s. 0d. per annum.

*Ion Exchangers in Organic and Biochemistry.* Edited by C. Calmon and T. R. E. Kressman. New York: Interscience Publishers Inc. 1957. Pp. xii + 761. $15.00.

*The Science of Color.* Optical Society of America. Committee on Colorimetry. New York: Thomas Y. Crowell Co. 1953, reprinted 1954. Pp. xiii + 385. 25 colour plates. $7.00.

*The Law of Copyright.* J. P. Eddy. London: Butterworth & Co. (Publishers) Ltd. 1957. Pp. xvii + 356. 35s. 0d.

*Textile Chemistry and Testing in the Laboratory.* R. W. Padgett. Minneapolis: Burgess Publishing Co. 1956. Pp. ii + 147.

*The Industrial Chemistry, Properties, and Application of Silicones.* C. E. Reed. Philadelphia: American Society for Testing Materials. 1956. Pp. iv + 47 $1.50.

*Forschungsberichte des Wirtschafts- und Verkehrsministeriums Nordrhein-Westfalen. No. 442. Die Auswirkungen der Trocknungsart nassgesponnener Leinengarne auf deren Verarbeitungswirkungsgrad sowie auf die Festigkeits- und Dehnungseigenschaften der Garne und Gewebe.* W. Rohs, H. Griese, and W. Lauer. Cologne: Westdeutscher Verlag. 1957. Pp. 18. DM 6.50.

*Register of Fellows and Associates, 1956.* London: Royal Institute of Chemistry. Pp. xx + 557.

*Farbnamenlexikon von A-Z.* G. Seufert. Göttingen: Musterschmidt-Verlag. 1955. Pp. 305.

*Handbuch für Textilingenieure und Textilpraktiker. Fachteil T14. Mechanisch-technologische Textilprüfungen.* E. Wagner. Wuppertal-Elberfeld: Dr. Spohr-Verlag. 7th auflage. 1957. Pp. viii + 240. DM 15.00.

*Précis des Matières Colorantes Synthétiques.* Tome II. *Matières Colorantes.* H. Wahl. Paris: Presses Universitaires de France. 1957. Pp. iv + 399. 2,800 Francs.

# Abstracts from British and Foreign Journals and Patents

*The titles of abstracts may be modified. Abbreviations of names of firms are listed in* J.S.D.C., **68**, *23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index*

## I—PLANT; MACHINERY; BUILDINGS

PATENTS

### Continuous Treatment of Cloth in Rope Form

Wiesner-Rapp Co. *USP* 2,736,183

A machine in which the cloth passes through several baths. It is readily "threaded-up" and the tension is separately controlled at various stages of processing.

C.O.C.

### Mounting for the Doctor Roller of a Screen Printing Machine

Zimmer's Erben KG. *BP* 773,562

Bending of the doctor roller is prevented by use of a non-rotating support in which is a recess of circular cross section open on the printing side. The support surrounds the roller with clearance on at least a part of its length.

C.O.C.

### Hosiery Setting and Dyeing

E. Bellman *BP* 774,797

Hose of polyamide yarns mounted on forms is continuously and individually introduced into separate treatment chambers which are then sealed enabling hot dye liquor to be sprayed into the chamber thereby giving combined setting and dyeing. Several types of machine construction are shown, the chambers being sealed either manually or automatically.

G.E.K.

### Garment Press

A. Humer *BP* 772,552

Shrinking Fabric with Improvement in Handle (X p. 412)

## II—WATER AND EFFLUENTS

### Effect of Starch Substitutes on Textile Wastes

J. A. McCarthy

*Sanitalk*, **4** (1), 23–5 (1955); *Water Pollution Abstr.*, **29**, 248 (1956)

Replacement of some of the starch by substitutes lowers the biological oxygen demand of wastes containing them and with mixtures of starch and carboxymethylcellulose exertion of the B.O.D. is retarded.

C.O.C.

### Production of Activated Anthracite for Purification of Waste Waters of the Aniline-dye Industry

L. A. Kul'skii, A. M. Koganovskii, K. E. Makhorin, E. M. Kalinichuk, V. M. Chertov, and E. I. Dikolenko

*Ukrainian Chem. J.*, **23**, 117–121 (Jan. 1957)

The activation of anthracite at 800–950°c. by steam and a mixture of the combustion gases of benzene with vapour is studied on a laboratory and a semi-industrial scale. 1 g. of anthracite of medium porosity adsorbs 150–200 mg. phenol and up to 300 mg. Methylene Blue. Under the conditions used the activated anthracite is superior to activated carbon.

G.J.K.

## III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

### Sheep-branding Fluids

M. Lipson

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 376–E 383, E 594–E 596 (1955)

A review has been made of the financial loss to the wool textile industry resulting from the use of non-scourable sheep-branding fluids. Progress of research on overcoming the problem is reported. Wide use of lanolin base emulsion branding fluids has greatly improved the situation, but limited use of harmful materials still causes difficulties in processing. Si-ro-mark branding fluid which has been developed is applicable to wet sheep. Extended use of this product should result in the total elimination of the problem from the Australian wool clip.

P.G.M.

### Recovery of Lanolin from Wool Scour Liquors

C. A. Anderson

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 363–E 375, E 592–E 594 (1955)

The aeration process of Evans and Ewers (*Australian J. Appl. Sci.*, **4**, 552 (1953)) for recovery of lanolin from wool scour liquors has been tested on an industrial scale. With the particular type of flotation cells used, overall recoveries of 40% were obtained; however, this yield was considerably reduced by the use of excessive amounts of soap in scouring. In laboratory tests recovery was reduced from 55 to 15% when soap content was increased from 0·05 to 0·4%. Churning of hot scour liquors also caused a drop in recovery. The addition of 0·1% $CaCl_2$ to liquors of high soap content increased recovery from 26·8 to 40·2%. The effect of age of scour liquors on recovery was also investigated. Liquors from daggy and similar dirty wools decomposed rapidly and recovery was reduced after relatively short storage periods. A commercial plant was installed at a cost of £(A)4,000. The plant is producing 750 lb./week of lanolin at a production cost of 6·25d./lb.

P.G.M.

### Stable Ultraviolet Absorbers

G. M. Gantz and W. G. Sumner

*Text. Research J.*, **27**, 244–251 (March 1957)

The structural requirements of an ultraviolet absorber are discussed, and data are presented on a series of substituted benzophenones which are effective and stable absorbers. Possible applications include protective finishes for fabrics degraded by ultraviolet radiation and stabilisers for improving the light fastness of certain dyes.

J.C.F.

### Preservation of Carboxymethylcellulose Solutions

L. G. Anderson

*Svensk Papperstidning*, **60**, 293–298 (30 Apl. 1957)

CMC solutions, although normally fairly resistant to microbiological attack, may be degraded by certain organisms. A series of preservatives has been investigated to determine their inhibitive effect on such micro-organisms. Phenylmercuric nitrate, a mixture of phenylmercuric acetate and oxine, and non-mercurial compounds such as oxine, dichlorophene, sodium pentachlorophenoxide + borax (50 : 50), etc., were found to give complete protection.

K.W.

### Kinetics and Mechanism of Iodination of Aromatic Aminosulphonic Acids in Aqueous Solutions

A. N. Kurakin and E. A. Shilov

*Ukrainian Chem. J.*, **23**, 31–53 (Jan. 1957)

The kinetics and mechanism of iodination of *N*-alkylsulphanilic and -metanilic acid derivatives are investigated in the presence and absence of phosphate buffer. The sulphonate ion is the reacting entity. Two iodine–tertiary amine complexes are postulated as transition states. Neutral salts have no effect upon the reaction.

G.J.K.

### Photochemical Isomerisation of Methylated Derivatives of Dithizone and related Compounds

P. S. Pel'kis and R. G. Dubenko

*Ukrainian Chem. J.*, **23**, 64–68 (Jan. 1957)

The kinetics of the isomerisation of the *trans* isomer of *S*-methyl derivative of dithizone and the *cis* isomer of di-*o*-tolylthiocarbazone in benzene solution under the influence of sunlight and artificial light with and without piperidine as catalyst are studied and found to represent a first-order reaction. The rate of isomerisation of the *cis* isomer of *S*-methyl-*o*-tolylthiocarbazone into the *trans* configuration is much greater than the reverse process.

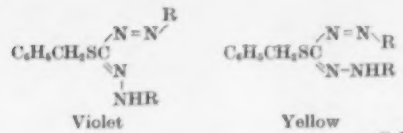
G.J.K.

### Isomeric Transformations of Benzyl Derivatives of Dithizone and related Compounds

P. S. Pel'kis and R. G. Dubenko

*Ukrainian Chem. J.*, **23**, 69–71 (Jan. 1957)

The synthesis of the benzyl derivatives of dithizone, 1:5-di-(*o*-tolyl)- and 1:5-di-(*o*-phenoxyphenyl)-thiocarbazone, is carried out and their isomerisation under the influence of light is investigated. By analogy to the methyl derivatives the benzyl compounds are claimed to possess the following configurations—



Violet Yellow

G.J.K.

PATENTS

**Dispersing Agents from Polyalkylene Oxide Waxes**
Boehme Fettchemie *BP* 772,454

Condensing a carboxylic acid of basicity > 2, e.g., citric acid, with a polyalkylene oxide wax, e.g., one of mol. wt. 9300, and simultaneously or subsequently with a fatty alcohol of > 5 C, a naphthene alcohol, resin alcohol, *cyclo*hexyl alcohol (which may be alkylated), alkyl phenol or alkyl naphthol (Alk of 3–18 C), e.g., octadecyl alcohol, yields a waxy water-soluble product useful as an emulsifying agent. The emulsions obtained are very stable to neutral, acid or alkaline electrolytes of all types.
C.O.C.

**Dispersions Stable in Presence of Electrolytes**
Boehme Fettchemie *BP* 773,508

Use as dispersing agents of the condensates obtained from polyalkylene oxide waxes of mol. wt. > 1000, phosphoric acids or anhydrides and chlorides of phosphorous, aliphatic, *cyclo*aliphatic or aliphatic-aromatic compounds of > 5 C, yields dispersions stable in presence of electrolytes. C.O.C.

**Water-soluble *N*-Methylol Polypyrrolidones as Finishing Agents**
Arnold, Hoffman & Co. *USP* 2,734,004

Products soluble in hot water and free from undesirable cross-linking are obtained by treating polypyrrolidone with HCHO in presence of a monohydroxycarboxylic acid, preferably aq. glycollic or lactic acid. Their aqueous solutions are used as textile finishes which on drying are fast to boiling aqueous soap solution. C.O.C.

**Water-repellent Finish**
Commonwealth Engineering Co. *USP* 2,734,005

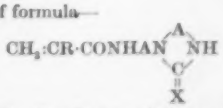
Higher fatty acid esters of dextran are good water-repellent agents which resist repeated washings. They also impart dimensional stability. They are applied from either aqueous dispersion or solution in a non-polar solvent, e.g., carbon tetrachloride or toluene. C.O.C.

**Water-repellent Silicone Compositions**
General Electric Co. *BP* 773,526

A mixture of (*a*) an organopolysiloxane containing no H bonded to Si and whose organic groups are bonded to Si by C at least 50% of them being $CH_3$ and the remainder $CH_3$, $C_2H_5$ or $C_6H_5$, (*b*) an incompletely condensed urea–aldehyde or melamine–aldehyde resin, and (*c*) an inorganic or organic salt of Ti, Zr, Sn, Pb, Al, Zn, Ni or Sr, is used for rendering textiles, paper, etc. water-repellent. The finish given is resistant to a large number of launderings or dry-cleanings. C.O.C.

**Amides— Finishing Agents**
Rohm & Haas *BP* 772,295

Compounds of formula—



(R = H or $CH_3$; X = O or S; A = alkylene of 2–3 C; there are at least 2 C between each pair of N atoms) readily form polymers and copolymers which are useful as textile and paper finishes, in coating compositions and for incorporation in viscose and cellulose acetate spinning dopes.
C.O.C.

**Brominated Phosphonate Polymers as Flame-proofing Agents**
Victor Chemical Works *USP* 2,735,789

Cloth treated with a solution of a brominated partial polymer of a dialkenyl monochloromethanephosphonate and then cured at moderate temperatures is given a flame-proof finish which is fast to repeated washings. The brominated partial polymer is obtained by dissolving a di-(β*γ*-unsaturated) alkenyl monochloromethanephosphonate, incompletely polymerising it, separating the partial polymer and then dissolving and brominating it.
C.O.C.

**Softening Agents**
Ciba *BP* 773,336

Softening agents especially suitable for use on regenerated cellulose or polyacrylonitrile fibres are obtained by condensing 1 mol. of a polyalkylene polyamine containing > 2 amino groups with at least 2 mol. of a non-aromatic monocarboxylic acid containing a hydrocarbon radical of 8–40 C and with at least 1 mol. of acrylonitrile or methacrylonitrile or acrylic or methacrylic amide which may contain substituents, or of a corresponding *p*-halogenopropionic acid derivative. Thus 0·25 mol. acrylonitrile are combined with 0·25 mol. diethylenetriamine, 0·5 mol. stearic acid is then added and the whole heated in an atmosphere of N at 160–165°C. for 4–5 hr. The product is then treated with acetic acid to yield a paste which can be diluted with water to a clear solution.
C.O.C.

**Hair Waving Composition**
Gillette Co. *BP* 773,559

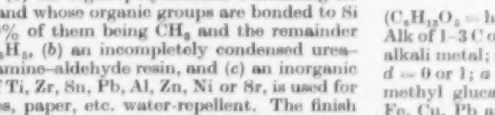
The hair is treated with a compound which splits the disulphide bond, e.g. ammonium thioglycollate, and then set with a compound containing at least one olefinic or acetylenic bond not forming part of an aromatic ring and which is capable of reacting with mercaptan groups, e.g. maleic, fumaric or acrylic acid or crotonaldehyde.
C.O.C.

**Solvents or Softeners for Polyesters of Terephthalic Acid and Aliphatic Dihydroxy Compounds**
FH *BP* 772,600

Aromatic or partly hydrogenated aromatic aldehydes or ketones, and aldehydes of aromatic heterocyclic compounds containing N as the hetero atom, are good solvents for poly(ethylene terephthalate) etc. Thus *cyclo*hexenylmethylketone dissolves the polyester at 150°C. while *o*-nitrobenzaldehyde dissolves it at 50°C., 14 other solvents are listed which act at temperatures between these two.
C.O.C.

**Sequestering Polyvalent Metal Ions of Atomic Number greater than 24**
Atlas Powder Co. *BP* 772,820

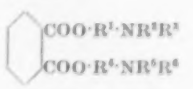
Compounds of formula—



($C_6H_{13}O_5$ = hexanepentol residue of a hexitylamine; R = H, Alk of 1–3 C or hydroxyalkyl of 2–3 C; Y = H or monovalent alkali metal; *n* = 1 or 2; *a* = 0 or 1; *b* = 0, 1 or 2; *c* = 0 or 1; *d* = 0 or 1; *a* + *b* + *c* + *d* = 2), e.g. *N*-methyl-*N*-carboxymethyl glucamine) are excellent sequestering agents for Fe, Cu, Pb and other metals of atomic number > 24.
C.O.C.

**Gas Fading Inhibitors**
Celanese Corp. of America *USP* 2,736,631

Compounds of formula—



($R^1$ and $R^4$ = same or different alkylene; $R^2$, $R^3$, $R^5$ and $R^6$ = same or different Alk), e.g. di-(diethylaminoethyl) phthalate, are gas fume fading inhibitors which may be either incorporated in the fibre substance before the fibre is formed, or be padded into the fibres, or added to the dye-bath. C.O.C.

**Dye-fixing Agents**
Arnold, Hoffman & Co. *USP* 2,736,749

Direct dyeings on cellulose are rendered faster to washing by treating them with the product obtained by reacting aqueous guanidine with formaldehyde at < 80°c., the reaction period being > 15 min. at 80°c. and about 20–24 hr. at 20–35°c. C.O.C.

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Modern Additions to Dry-cleaning Solvents and their Action (VII p. 408)

# IV— RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

### Synthesis of Dye Intermediates
R. Oda
*Yûki Gôsei Kagaku Kyôkaishi*, **14**, 205–208 (1956)
Review. 10 references. C.O.C.

### Colour Reactions of Titanium with Phenols
S. Ya. Shnaiderman
*Ukrainian Chem. J.*, **23**, 92–96 (Jan. 1957)
The colour reactions of titanium with phenols in acid conditions are investigated spectrophotometrically. G.J.K.

### Dinitrobenzenesulphonic Acids and their Ethers
A. I. Kiprianov and A. I. Tolmachev
*J. Gen. Chem. U.S.S.R.*, **27**, 486–491 (Feb. 1957)
The preparation of a series of dinitro- and one mono-nitrotrifluoromethylbenzenesulphonic acid and also that of certain of their alkyl ethers are described. G.J.K.

### Orientation in the Aromatic Series. I— Sulphonation of *m*-Toluenesulphonic Acid
A. A. Spryskov and T. I. Yakovleva
*J. Gen. Chem. U.S.S.R.*, **27**, 239–244 (Jan. 1957)
The effect of different sulphonating agents, of time of reaction and of temp. upon the sulphonation of *m*-toluene-sulphonic acid is investigated. Of the two isomers (1:2:5 and 1:3:5) the 1:3:5 derivative is the more stable. G.J.K.

### Nitration of 1-*n*-Butylnaphthalene
S. I. Sergievskaya and T. S. Safonova
*J. Gen. Chem. U.S.S.R.*, **27**, 421–428 (Feb. 1957)
Nitration of 1-*n*-butylnaphthalene under different conditions yields primarily the 4-nitro derivative, although the 4:5-dinitro compound and the 2:4:5-trinitro derivative are formed under more rigorous conditions. The structure of the first two compounds is proved unambiguously. G.J.K.

### 4-Amino-1-*n*-butylnaphthalene and its Reactions
S. I. Sergievskaya and T. S. Safonova
*J. Gen. Chem. U.S.S.R.*, **27**, 428–431 (Feb. 1957)
The preparation of the 4-amino-(its hydrochloride, and acetyl- and benzoyl derivatives)-1-*n*-butylnaphthalene and the conversion of the 4-amino compound to the 4-chloro-, 4-iodo- and 4-carboxylic acid derivatives are described. G.J.K.

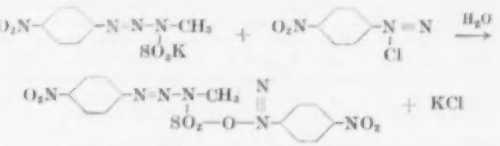
### Dyes of the Benzene and Naphthalene Series
H. Hiyama
*Yûki Gôsei Kagaku Kyôkaishi*, **14**, 209–212 (1956)
Review. 22 references. C.O.C.

### Synthesis of Azo Dyes from 4:4′-Diaminodiphenylurea
D. F. Kutepov and Z. G. Yukolova
*J. Gen. Chem. U.S.S.R.*, **27**, 200–201 (Jan. 1957)
A direct brown prepared from tetrazotised 4:4′-diaminodiphenylurea with one molecule of γ-acid and one of salicylic acid is not inferior to the corresponding benzidine derivative. G.J.K.

### Azo Derivatives of Benzothiazole— II
A. I. Kiprianov and I. P. Fedorova
*Ukrainian Chem. J.*, **23**, 59–63 (Jan. 1957)
2:2′-Dimethyl-5:5′-azobenzthiazole and a series of 2-methyl-5- and 2-methyl-6-arylazobenzthiazoles are prepared and their absorption spectra in alcoholic solution are determined. G.J.K.

### Structure and Transformations of *N*-nitrotriazens. II— Methylaryl-*N*-nitrotriazens
N. M. Baranchik, I. V. Grachev, and D. Z. Zavel'skii
*J. Gen. Chem. U.S.S.R.*, **27**, 117–126 (Jan. 1957)
By condensing diazonium compounds with *N*-methylnitramine a new series of *N*-nitrotriazens are produced, but the compounds are limited due to the fact that methylnitramine only condenses with active diazonium compounds. Methylaryl-*N*-nitrotriazens are important because they can couple with β-naphthol in neutral alcoholic solution due to the presence of the electrophilic nitro group attached to the nitrogen of the triazene system. On heating in dilute $H_2SO_4$ decomposition of the methylaryl-*N*-nitrotriazen rapidly produces $N_2O$ from *N*-methylnitramine whereas $N_2$ is evolved more slowly from the aryldiazonium compound. The presence of nitro groups in the aryl nucleus hinders the hydrolysis of the methylaryl-*N*-nitrotriazens. G.J.K.

### Reaction of Diazo Compounds with Sulphamic Acid and its Derivatives. IV— Diazonium Salts of Methylaryltriazen-*N*-sulphonic Acids
D. Z. Zavel'skii and L. A. Lishnevskaya
*J. Gen. Chem. U.S.S.R.*, **27**, 388–398 (Feb. 1957)
Diazonium salts of aryl-1-methyl-3-triazene sulphonic acids (D8) are prepared (*a*) from methylsulphamic acid and nitro-aryl-diazo compounds under acidic conditions, and (*b*) by a salt exchange reaction of diazonium chloride and the potassium salts of aryl-1-methyl-3-triazene-sulphonic acid—



The properties and decomposition reactions of D8 are studied and it is shown that their stability decreases with increasing electrophilic nature of the substituents in the nucleus of the diazo residue. G.J.K.

### Benzalazen and its Derivatives. I— Action of HCl on Benzalazen and its Derivatives
B. M. Bogoslovskii and T. I. Yakovenko
*J. Gen. Chem. U.S.S.R.*, **27**, 159–168 (Jan. 1957)
A number of new mono- and di-hydrochlorides of symmetrical benzaldehyde-azens have been prepared by the interaction of dry HCl on the azen. The structure of these compounds has been investigated spectroscopically and the effect of substituents on the colour and on the mono- and di-salt formation are explained on the basis of modern electronic theory. G.J.K.

### Phenylmethylaminomethylcarbinol
G. M. Borodina
*J. Gen. Chem. U.S.S.R.*, **27**, 281–283 (Jan. 1957)
Phenylmethylaminomethylcarbinol (I) is separated into *d*- and *l*-forms. The two forms of I are nitrated; the nitro-compounds so formed hydrogenated using Pd-charcoal catalyst to give the corresponding amino compounds which are diazotised and coupled with β-naphthol; the azo dyes of these two forms result. G.J.K.

### Silicon Analogue of Crystal Violet— II
F. Brandmair and U. Wannagat
*Z. anorg. u. allgem. Chem.*, **288**, 91–100 (1956): *Chem. Abs.*, **51**, 6530 (10 May 1957)
The spectral distance between the corresponding absorption maxima in the spectra of $R_3CCl$ ($R = p\text{-}(CH_3)_2NC_6H_4$) and $C_6H_5CCl$ has no parallel in the spectra of $R_2SiCl$ and $C_6H_5SiCl$. Using a KBr prism measurements were extended to the range of 2–16 μ. The frequencies attributed to the Si-Cl band in the spectra of $R_2SiCl$ and $C_6H_5SiCl$ differed only slightly from one another; surprisingly, these frequencies were higher than the corresponding frequency in the spectrum of $(CH_3)_2SiCl$, indicating a lesser degree of polarity of the Si-Cl bond in the aromatic compounds. Debyeograms of $R_2SiCl$ and $R_3CCl$ were essentially different; this confirms the important difference between the spatial structures of the two compounds. A non-polar structure without an Si double bond was attributed to $R_2SiCl$ in contrast to the quinoid structure attributed to Crystal Violet. C.O.C.

### Photoreduction of Eosin in the Bound State
J. S. Bellin and G. Oster
*J. Am. Chem. Soc.*, **79**, 2461–2464 (20 May 1957)
The spectral and photochemical properties of Eosin Y (2′:4′:5′:7′-tetrabromofluorescein) bound to polyvinylpyrrolidone differ considerably from those of the free dye. The bound dye exhibits self-quenching of fluorescence at much lower concentrations than does the free dye, the reverse being true for self-quenching of phosphorescence in rigid media. Furthermore, *p*-phenylenediamine which quenches the phosphorescence of the free dye does not affect the bound dye. Nitrobenzene retards photoreduction of free Eosin but inhibits that of bound Eosin, the duration of the inhibition period being proportional to the concentration of the inhibitor. *p*-Phenylenediamine both

inhibits and retards photoreduction of bound Eosin while it only retards that of the free dye. For small dye concentrations the quantum yield of photoreduction of bound Eosin increases with increasing dye concentration, the opposite being true for the free dye. These rate studies suggest that a bound dye molecule in the first electrically excited state rapidly exchanges energy with a bound dye molecule in the ground state to produce a long-lived excited species which reacts with the reducing agent (ascorbic acid was used). A mechanism is proposed for dye-sensitisation in silver halide photography based on photoreduction of bound dye. C.O.C.

**Irreversible Photobleaching of Solutions of Fluorescent Dyes**

M. Imamura and M. Koizumi

**II— Alcoholic Solutions of Eosin *in vacuo***

*Bull. Chem. Soc. Japan*, **29**, 899–903 (Nov. 1956)

The primary process of photobleaching of Eosin in evacuated ethanol, *iso*-propanol, and *n*-butanol is dehydrogenation of the alcohol by excited dye yielding aldehyde and semiquinone radical. Introduction of oxygen causes induction periods and the formation of acids and $H_2O_2$ by the reaction of alkoxide and semiquinone radicals with $O_2$. The results are readily explained by the mechanism of Bolland and Cooper (*Proc. Roy. Soc.*, **225**, 405 (1954)).

**III— Photoreaction of Eosin in Aqueous Alcoholic Solution *in vacuo***

*Ibid.*, 913–918

Eosin is debrominated to uranine when irradiated in aqueous ethanol, the rate of disappearance of Eosin increasing with increasing concn. of ethanol to a constant value at 20% by vol. A.J.

**Cyanine Dyes. I— Absorption of Cyanines derived from Pyranothiazole and Thiopyranothiazoles**

R. H. Sprague

*J. Amer. Chem. Soc.*, **79**, 2275–2281 (5 May 1957)

Three new thiazole bases have been obtained by condensing thioacetamide with 3-bromotetrahydro-4-pyrone, 3-bromo-tetrahydro-1-thio-4-pyrone and 2-bromotetrahydro-1-thio-3-pyrone. Their quaternary salts give rise to cyanine dyes some of which are powerful photographic sensitisers. The absorptions of these dyes are strikingly different from those of similar dyes from benzothiazole and tetrahydrobenzothiazole. C.O.C.

**Relationship between Constitution and Photographic Properties of Organic Dyes**

O. Riester

*Mitt. Forschungslab. Agfa Leverkusen-München*, **1**, 44–55 (1955): *Chem. Abs.*, **51**, 6405 (10 May 1957)

Dyes having the characteristic group—

$$\rangle N\text{-}(\overset{|}{C}:\overset{|}{C}\text{-})_n C{:}(\overset{|}{C}\text{-}\overset{|}{C})_m{:}\overset{\oplus}{N}\langle$$

which are photographic sensitisers and those having the group—

$$\rangle N\text{-}(\overset{|}{C}:\overset{|}{C}\text{-})_n N{:}(\overset{|}{C}\text{-}\overset{|}{C})_m{:}\overset{\oplus}{N}\langle$$

which are predominantly desensitisers are compared in terms of electronic theory. Cyanines containing cyano as a substituent in the first methin group of the bridge can be got from intermediates such as 2-cyanomethylene-3-methylbenzothiazoline which is prepared by treating a 3-methyl-2-methylthiobenzothiazolium salt with methylcyanoacetate, and hydrolysing with (spontaneous) decarboxylation. Treatment of the 3-methyl-2-methylthiobenzothiazolium salt with nitromethane yielded 3-methyl-2-nitromethylenebenzothiazoline, which on hydrolysis yielded trimethincyanines. C.O.C.

**Colour and Chemical Constitution of Dyes**

T. Mine

*Yûki Gôsei Kagaku Kyôkaishi*, **14**, 185–189 (1956)

Review, 13 references. C.O.C.

**Effect of Environment upon Colour of Organic Unsaturated Compounds**

M. Koizumi and N. Mataga

*Yûki Gôsei Kagaku Kyôkaishi*, **14**, 181–185 (1956)

Review, 27 references. C.O.C.

**Cyanine Dyes. X— *mero*Cyaninocarbocyanine Dyes**

M. V. Deichmeister, I. I. Levkoev, and E. B. Lifshits

*J. Gen. Chem. U.S.S.R.*, **27**, 202–215 (Jan. 1957)

Various *mero*cyaninocarbocyanine dyes based upon the thiazol-4-ine and benzothiazole systems have been synthesised, and their absorption spectra studied. The increase in the length of the polymethine chain, particularly in the *mero*cyanine portion of the molecule, causes a decrease in the bathochromic displacement, whereas a methyl- or ethyl group attached to a chain carbon atom produces a hypsochromic shift. A deepening of colour is observed when the basicity of the N-containing heterocyclic residue of the cyanine part of the *mero*cyaninocarbocyanine molecule is increased. G.J.K.

***mero*Cyanine Dye Derivatives of Rhodamine. VII— Reaction Products of 3-Ethyl-5-(3′-ethyl-6′-diethylaminobenzthiazolinylidene-2′-ethylidene)-thiazoli-denethion-(2)-one-(4) with dimethylsulphate and their Transformations**

Z. P. Sŭtnik and L. D. Zhilina

*J. Gen. Chem. U.S.S.R.*, **27**, 215–227 (Jan. 1957)

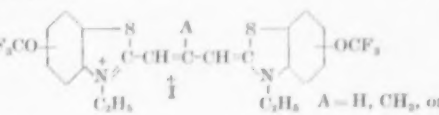
The synthesis of new quaternary salts of the dimethyl-*mero*cyanine dye derivatives based on 2-methylmercaptothiazolinone-(4) with dialkylamino substituents is described and from the study of their absorption spectra the effect of the quaternary ammonium salt grouping is shown to produce an appreciable hypsochromic shift. G.J.K.

**Cyanine Dyes containing Fluorine. V— Synthesis of Cyanine Dyes from 5- and 6-Trifluoromethoxybenzothiazole**

L. M. Yagupol'skiĭ and V. I. Troitskaya

*J. Gen. Chem. U.S.S.R.*, **27**, 518–526 (Feb. 1957)

2-Methyl-5-, and 2-methyl-6-trifluoromethoxybenzothiazoles are prepared. Eight carbocyanine dyes containing the trifluoromethoxy groups in 5 and 6 positions of the benzothiazole ring are obtained as their quaternary iodides—




It is found that the spectra of the salts are not appreciably affected by the presence of the $-O\text{-}CF_3$ group. G.J.K.

**Free-radical Salts of Halogenated Quinones**

H. Kainer, D. Bijl, and A. C. Rose-Innes

*Nature*, **178**, 1462–1463 (29 Dec. 1956)

The salts formed by the action of sodium iodide on tetrahalogenated quinones are shown, by the paramagnetic resonance method, to be the sodium salts of semi-quinone radicals. A.J.

**Dyes of the Anthracene Series**

*Yûki Gôsei Kagaku Kyôkaishi*, **14**, 212–216 (1956)

Review, 10 references. C.O.C.

**Remarks on the Structure of Aniline Black on the Basis of Ultraviolet Absorption Spectra**

T. Urbanski and K. Szye-Lewanska

*Bull. Acad. polon. sci., Classe III*, **4**, 775–781 (1956): *Chem. Abs.*, **51**, 7144 (25 May 1957)

The ultraviolet spectra of Emeraldine and Aniline Black dissolved in $H_2SO_4$ are similar to that of dianilinoquinone. The spectra of phenazine, safranine and Phenol Blue differ considerably. C.O.C.

**Anthraquinone Vat Dyes**

A. Tundo

**I— Acylaminoanthraquinone from Furan-2:5-dicarboxylic Acid**

*Ann. chim. (Rome)*, **46**, 1183–1188 (1956)

Condensation of substituted 1-aminoanthraquinones with 2:5 $C_4H_2O(COCl)_2$ (I) gives diamides analogous to the Paradone dyes prepared from $p\text{-}C_6H_4(COCl)_2$. The products have absorption maxima between 420 and 460 mμ. in nitrobenzene and excellent fastness to light and alkali. The following *NN′*-di-1-anthraquinonyl-2:5-difurandicarboxamides substituted in the anthraquinone rings are prepared by this method: no subst., yellow; 2:2′:4:4′-$Br_4$, khaki; 4:4′-$Cl_2$, yellow; 4:4′-$(OH)_2$, reddish brown; 4:4′-$(OCH_3)_2$, orange-red; 4:4′-$(C_6H_5S)_2$, bright red;

4:4′-(NHSO$_3$C$_6$H$_5$)$_2$, orange-red; 5:5′-(OH)$_2$, golden yellow; 5:5′-(NH$_2$)$_2$, reddish brown; 5:5′-(NHBz)$_2$, yellow; 8:8′-(OH)$_2$, golden yellow; 8:8′-(NH$_2$)$_2$, maroon; 8:8′-(NHBz), yellow; 2:2′:4:4′-Bz$_2$(OH)$_2$, maroon; 4:4′:5:5′-(OH)$_4$, reddish maroon. Similarly, using *p*-C$_6$H$_4$(COCl)$_2$ (II) instead of (I) *NN′*-di-1-anthraquinonylterepthalamide (yellow) (substituted in the anthraquinone rings) and its 4:4′-(SC$_6$H$_5$)$_2$ (bright red) and 4:4′-(NHSO$_2$C$_6$H$_5$)$_2$ (orange red) derivatives, were prepared.

### II— Acylaminoanthraquinones from Furan-2:5-dicarboxylic Acid and Terephthalic Acid

*Ibid.*, 1189–1193

Condensing 1-amino-2:4-dibromoanthraquinone with arylamines in butanol yields a green insoluble product besides the blue 1-amino-2-bromo-4-arylaminoanthraquinone (III) soluble in chlorobenzene. Boiling III dissolved in chlorobenzene with I or II yields *NN′*-bis(2-bromo-4-arylamino-1-anthraquinonylterephthalimide (aryl group, colour on rayon) C$_6$H$_5$, violet; 1-naphthyl, violet; 2-naphthyl, bluish violet; 4-biphenylyl, bluish indigo; *p*-CH$_3$C$_6$H$_4$, bluish violet; *p*-CH$_3$OC$_6$H$_4$, bluish violet; *p*-C$_2$H$_5$OC$_6$H$_4$, blue; *p*-ClC$_6$H$_4$, violet; and the following *NN′*-bis(2-bromo-4-arylamino-1-anthraquinonyl)-2:5-furandicarboxamides: C$_6$H$_5$, violet; 1-naphthyl, violet; 2-naphthyl, bluish violet; *p*-CH$_3$C$_6$H$_4$, violet; *p*-CH$_3$OC$_6$H$_4$, violet; *p*-C$_2$H$_5$OC$_6$H$_4$, bluish indigo. Derivatives of I and II differ little in absorption, but those of I vat much more readily; under the drastic dyeing conditions necessary some derivatives of II give bluer dyeings.

### III— Acylaminoanthraquinones from Furan-2:5-dicarboxylic Acid and Terephthalic Acid

*Ibid.*, 1194–1198:
*Chem. Abs.*, **51**, 7106 (10 May 1957)

The following dyes all very fast to light and alkali were prepared by the above methods. *NN′*-bis(2-bromo-4-arylamino-1-anthraquinonyl)terephthalimides: *o*-CH$_3$C$_6$H$_4$, violet blue; *m*-CH$_3$C$_6$H$_4$, blue; *o*-ClC$_6$H$_4$, violet; *m*-ClC$_6$H$_4$, violet; *o*-CH$_3$OC$_6$H$_4$, blue; *m*-CH$_3$OC$_6$H$_4$, blue; 2:5(CH$_3$O)$_2$C$_6$H$_3$, intense blue; 2:4-CH$_3$(Cl)C$_6$H$_3$, violet; 2:4:6(CH$_3$)$_3$C$_6$H$_2$, violet; *NN′*-Bis-(2-bromo-4-arylamino-1-anthraquinonyl)-2:5-furandicarboxamides: *o*-CH$_3$C$_6$H$_4$, violet; *m*-CH$_3$C$_6$H$_4$, violet; *m*-ClC$_6$H$_4$, violet; *o*-CH$_3$OC$_6$H$_4$, violet; *m*-CH$_3$OC$_6$H$_4$, intense violet; 2:5-(CH$_3$O)$_2$C$_6$H$_3$, blue-violet; 2:4-CH$_3$(Cl)C$_6$H$_3$, violet; 2:4:6-(CH$_3$)$_3$C$_6$H$_2$, violet. C.O.C.

### *iso*Phthaloylaminoanthraquinone Vat Dyes

Y. Takewawa and S. Kato

*Yûki Gôsei Kagaku Kyôkaishi*, **15**, 87–91 (1957):
*Chem. Abs.*, **51**, 7721 (25 May 1957)

The synthesis of five types of the above dyes is described, together with study of the relationship between hue, dyeing category, fastness to light and chemical constitution. C.O.C.

### Dyes of the Phenanthrene Series

S. Kato

*Yûki Gôsei Kagaku Kyôkaishi*, **14**, 217–220 (1956)
Review, 9 references. C.O.C.

### Dyes from Pyrene and other Polynuclear Hydrocarbons

Y. Abe and Y. Nagai

*Yûki Gôsei Kagaku Kyôkaishi*, **14**, 220–223 (1956)
Review, 41 references. C.O.C.

### Heterocyclic Dyes

M. Yokote

*Yûki Gôsei Kagaku Kyôkaishi*, **14**, 224–227 (1956)
Review, 31 references. C.O.C.

### Spectroscopic Studies on Dyes. IV— Fluorescence Spectra of Thioindigo Dyes

D. A. Rogers, J. D. Margerum, and G. M. Wyman

*J. Amer. Chem. Soc.*, **79**, 2464–2468 (20 May 1957)

The fluorescence spectra of eight thioindigo dyes dissolved in benzene were determined using the 546 m$\mu$. Hg-line for excitation. Each dye has a fluorescence band at a wavelength somewhat longer (usually about 35 m$\mu$.) than its first absorption maximum. Exposing the solutions to yellow light before measuring the spectra results in decreased fluorescence. The intensity of the fluorescence is proportional to the concentration of *trans*-isomer present. This behaviour, similar to that of stilbene, indicates that *trans*-thioindigo has a tightly-held coplanar structure, while the *cis*-isomer is non-coplanar. This agrees with the visible and infra-red absorption data. Addition of ethanol quenches the fluorescence. These dyes do not show fluorescence when dyed on cellulose. As ethanol and cellulose are chemically similar this suggests an analogy between the mechanism by which the fluorescence of these dyes is quenched by ethanol and that by which they promote the tendering of cellulose on exposure to light. C.O.C.

### Stilbene Derivatives

R. Asaoka, H. Suzuki, and H. Iida

*Rept. Govt. Chem. Ind. Research Inst. Tokyo*, **52**, 25–29 and iii–iv (Jan. 1957)

4:4′-Diaminostilbene-2:2′-disulphonic acid (I), an important intermediate for dyes and fluorescent brightening agents, is prepared by the reduction of 4:4′-dinitrostilbene-2:2′-disulphonic acid (II), which is obtained by the oxidation of *p*-nitrotoluene-*o*-sulphonic acid with sodium hypochlorite solution. However, recently an air oxidation process has been adopted for the preparation of II, and the relations between the yield of I, the purity, and the oxidising conditions have been investigated.

Twenty grams of sodium *p*-nitrotoluene-*o*-sulphonate is dissolved in 280 ml. of water and warmed. Then, 30–33 g. of 30% NaOH solution is added to the above solution at 50°c. with mechanical stirring, 0·4 g. of MnSO$_4$ or FeSO$_4$ also being added, as catalyst. Immediately after the addition, with mechanical stirring being continued, air is introduced into the mixture for 20 hr. at a velocity over 60 litres/hr., and at the same time the temperature is gradually raised to 60°c. The rate of stirring is maintained at 550 r.p.m. throughout the operation. The amount of the crude sodium salt of II obtained is 15 g., corresponding to 87% of the theoretical, and the yield of I (93% purity) is 10 g. The time of oxidation can be shortened to 10 hr. by increasing the air velocity to 200–210 litres/hr. and previously warming the air to 50°c.

Paper-chromatographic studies on I were carried out using a mixture of *n*-butanol, ethanol, and water (2 : 1 : 2), as solvent at 17–19°c., and the following $R_f$ values were found—

| | | |
|---|---|---|
| 4:4′-Diaminostilbene-2:2′-disulphonic acid | 0·32 | 0·40 |
| 4:4′-Diaminodibenzyl-2:2′-disulphonic acid | 0·36 | |
| *p*-Toluidine-*o*-sulphonic acid | 0·62 | |

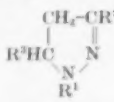
C.J.W.H.

PATENTS

### Fluorescent Compounds for Distinguishing Photographic Materials

Societa per Azioni Ferraria *BP* 772,916

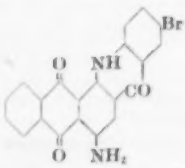
Compounds of formula—



(R$^1$, R$^2$ and R$^3$ = aromatic subst. or unsubst. radicals, one of which at least is attached to the pyrazoline ring by a methin chain), e.g., 1:5-diphenyl-3-styrylpyrazoline, incorporated in photographic films made from a cellulose derivative give strong fluorescence under ultraviolet radiation without affecting any photographic emulsion there may be on them. C.O.C.

### 4-Amino-5′-bromoanthraquinone-2:1-(*N*)-1′:2′-(*N*)-benzeneacridone

FBy *BP* 775,802

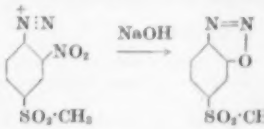



is produced by condensing 1:bromo-4:aminoanthraquinone 3:sulphonic acid with anthranilic acid; treating the product with the theoretical amount of Br in conc. H$_2$SO$_4$, 2–3% oleum or chlorosulphonic acid at 0–30°c., and finally ring-closing and desulphonating by heating the acid melt to 50–100°c. It may be used as a dye intermediate instead of the corresponding chloro compound. E.T.

**4-Alkylsulphonyl-2-Hydroxydiazo Compounds, and Metal-complex Monoazo Dyes Derived Therefrom**

Gy *BP* 774,885

4-Alkylsulphonyl-2-nitroanilines are diazotised in nitrosylsulphuric acid, and after drowning out in ice and water, excess mineral acid is neutralised, whereby the corresponding 1:2-diazoxide is produced. Thus—

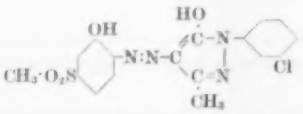


*BP* 774,886

The yield is improved by the presence of stabilisers, e.g., naphthalene-2:7-disulphonic acid.

*BP* 774,884

The diazoxides are coupled with suitable *ortho*-coupling hydroxy compounds, free of $SO_3H$ and COOH groups, to give monoazo compounds, whose 1:2 chromium complexes dye wool and nylon from neutral or weakly acid baths. Thus the diazoxide of the above example is coupled with an aq. NaOH soln. of 1-*m*-chlorophenyl-3-methyl-5-pyrazolone to give—

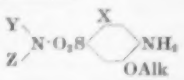


Boiling with ammonium chromosalicylate gives the metal complex, which dyes wool scarlet. E.S.

**Red, Blue, and Brown Metal(Cobalt)-complex Monoazo Dyes for Wool and Nylon**

BASF *BP* 775,163

Metal complexes containing 1 atom of Co to 2 mol. of monoazo compounds obtained by coupling a diazotised alkoxyanilinesulphonamide

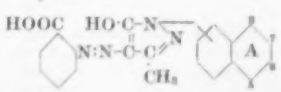


(X = H, Hal, Alk, or OAlk; Y = H, Alk, or phenyl; Z = H or Alk) with an *ortho*-coupling phenol, naphthol or enolic compound of the quinoline series, free of $SO_3H$ groups, dye wool and nylon from neutral or weakly acid baths. They are generally faster to light than the corresponding Cr complexes. Thus the monoazo compound 2-amino-4-methylanisole-5-sulphonamide→*p*-*iso*butylphenol is heated with cobalt acetate for 5 hr. at 130°C.; the product dyes wool bordeaux from a weakly acid bath. E.S.

**Yellow Metal(Chromium)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.**

S *BP* 775,216

The metal complexes containing 1 atom of Cr to 2 mol. of monoazo compounds—

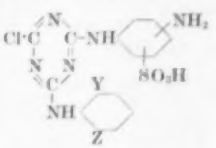


(the ring A may be hydrogenated in positions 5, 6, 7, and 8) dye wool nylon, etc., yellow. The unmetallised compounds are metachrome dyes. E.S.

**Yellow Monoazo Acid Dyes having a Cyanuric and a Pyrazolone Ring**

Ciba *BP* 775,308

Condensation products of cyanuric chloride—



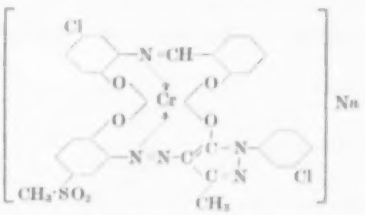
(Y and Z = H or substituents incapable of imparting solubility in water) are diazotised and coupled with suitable 5-pyrazolones to give yellow acid dyes applicable from weakly acid baths. Thus, the condensation product of cyanuric chloride with 1 mol. of aniline and 1 mol. of 1:3-diaminobenzene-4-sulphonic acid is diazotised and coupled with 1-*o*-chlorophenyl-3-methyl-5-pyrazolone to give a greenish yellow acid dye. E.S.

**Metal(Chromium)-complex Monoazo–Azomethin Dyes for Wool, Nylon, etc.**

Gy *BP* 775,385

The metal complexes containing 1 atom of Cr combined with 1 mol. of a suitable monoazo compound and 1 mol. of an azomethine compound, dye wool and nylon from a weakly acid bath a variety of shades. They are preferably made by boiling the 1:1 chromium complex of the monoazo compound with 1 mol. of the azomethine compound. Components are so chosen that the finished dye contains no $SO_3H$ or COOH groups, but the presence of sulphonamide, alkylsulphonyl, or acetylamino groups is advantageous in conferring solubility.

Thus the 1:1 Cr complex of the monoazo compound 2-amino-4-methylsulphonylphenol → 1-*m*-chlorophenyl-3-methyl-5-pyrazolone is boiled in alcohol with the azomethine from salicylaldehyde and 2-amino-4-chlorophenol in presence of sodium acetate. The product—

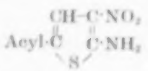


dyes wool yellow orange from an acetic acid bath. E.S.

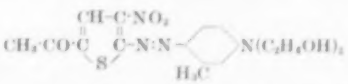
**Blue Disperse Azo Dyes from 2-Amino-3-nitro-5-acylthiophenes**

Eastman Kodak Co. *BP* 774,611

Thiophene derivatives—



are diazotised and coupled with coupling compounds of the aniline, tetrahydroquinoline, and benzomorpholine series to give blue and blue-green disperse dyes. Thus 2-chlorothiophene is stirred with $AlCl_3$ and $CH_3{\cdot}CO{\cdot}Cl$ in *n*-hexane at 40°C. to give 2-chloro-5-acetylthiophene. Nitration gives the 3-nitro compound, and refluxing with methyl alcoholic ammonia then gives 2-amino-3-nitro-5-acetylthiophene, which is diazotised in nitrosylsulphuric acid and coupled with *NN*-bis-β-hydroxyethyl-*m*-toluidine to give

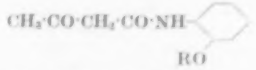


It dyes cellulose acetate, nylon, and Terylene dischargeable greenish blue. E.S.

**Yellow Disazo Pigments**

FH *BP* 774,676

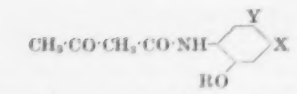
Tetrazotised 3:3′-dichlorobenzidine is coupled with 2 mol. of *N*-acetoacetylaniline derivatives



(R = Alk or Ar) to give bright yellow pigments. *N*-Acetoacetyl-*o*-anisidine, for instance, gives a greenish yellow.

*BP* 775,355

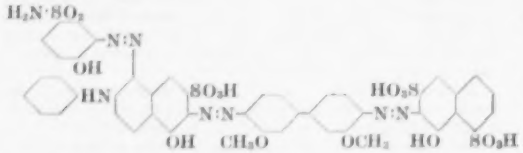
The same tetrazo compound with 2 mol. of *N*-acetoacetylaniline derivatives



(R = $CH_3$ or $C_2H_5$; X = Cl or OR; Y = H or OR; X must = OR if Y = H) to give similar yellow pigments. Thus, *N*-acetoacetyl-4-chloro-2:5-dimethoxyaniline gives a reddish yellow. E.S.

**Navy Blue Metal-complex Polyazo Direct Dyes**
S *BP* 775,320

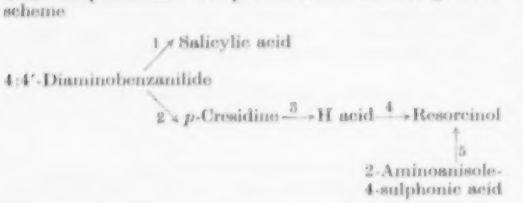
The polyazo dyes of *BP* 760,705 (*J.S.D.C.*, **73**, 67 (Feb. 1957)), providing sufficient water-solubilising groups are present, can be converted in substance into Cu or Ni complexes, which dye cellulose navy blue. Thus the trisazo compound



is boiled in aq. ammoniacal pyridine with $CuSO_4$ for 24 hr. and the soluble Cu complex is salted out. E.S.

**Metal(Copper and Nickel)-complex Polyazo Direct Dyes**
S *BP* 774,150

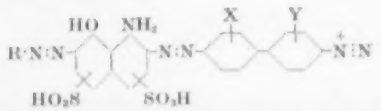
Resorcinol is coupled with two diazo compounds, one being derived from a metallisable dis- or tris-azo dye whose end component is an alkali-coupled 1:8-aminonaphtholsulphonic acid, and the other derived from an amine or aminomonoazo compound having a group capable of chelation in *ortho*-position to the amino group. The resulting polyazo compounds, which have 2–5 sulpho groups, are converted into copper or nickel complexes, which dye cellulose blue, olive, khaki, brown, and grey. Thus the pentakisazo compound formed according to the scheme




coupling 3 being made under alkaline conditions, and coupling 5 in presence of naphthalene-β-sulphonic acid and pyridine, is fused with $CuSO_4$ and sodium acetate at 107° c. to give a brownish khaki direct dye. E.S.

**Blue Trisazo Dyes for Leather**
CFM *BP* 774,612

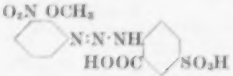
Diazodisazo compounds



(R = aryl of benzene or naphthalene series; X and Y = H, $CH_3$, or $OCH_3$) are coupled with a dihydroxynaphthalene-sulphonic acid to give blue dyes for leather, especially suède leather. Thus tetrazotised benzidine is coupled with 1 mol. of H acid in presence of mineral acid. One mol. of diazotised aniline is then added and coupling carried out in presence of soda ash. Finally the second diazo group of the benzidine residue is coupled with 2:8-dihydroxynaphthalene-6-sulphonic acid in presence of soda ash. The product dyes leather slightly reddish blue. E.S.

**Diazoamino Compounds for Azoic Dyes Developed by Neutral Steaming**
FBy *BP* 771,812

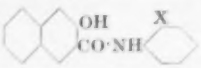
Diazotised derivatives of *m*- and *p*-nitroaniline substituted by 1 or 2 alkyl or alkoxy groups, or by 1 alkyl and 1 alkoxy group, give triazenes with *N*-alkyl-5(or 4)-sulphoanthranilic acid, which regenerate the diazo compound on neutral steaming. Mixtures of such triazenes with suitable coupling components can thus be used for producing azoic prints and can be used side by side with vat dyes. Thus a soln. of diazotised 2-amino-6-nitroanisole is run into a soln. in aq. sodium carbonate of *N*-ethyl-5-sulphoanthranilic acid, and the triazene



isolated by salting-out. It is mixed with a soln. in aq. NaOH of 3-hydroxy-2-naphtho-*o*-anisidide, suitably thickened, printed on cotton, and treated with neutral steam, to give a scarlet azoic dye. E.S.

**Bright Pink and Red Azoic Dyes**
FH *BP* 775,065

Azoic pinks and reds similar in brightness to the rhodamines are produced on cellulose by using 3-amino-4-methoxybenzamide as the diazo component and arylamides of 3:2-hydroxynaphthoic acid—



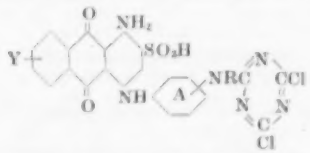
(X = H, Alk, or OAlk) as coupling components. E.S.

**bis-Triazinylamino Stilbene Fluorescent Brightening Agents**
Gy *BP* 773,152

Compounds of intensive blue fluorescence and very good affinity for cellulose are obtained by condensing 1 mol. of the condensate of 1 mol. 4:4′-diaminostilbene-2:2′-disulphonic acid and 2 mol. cyanic halide with 2 mol. of a primary aromatic amine of the benzene series which may have Cl, acylamino, alkyl, or alkoxy as substituents in the ring and with 2 mol. of secondary *N*-methyl or *N*-ethyl ethanolamine. Thus cyanuric chloride is condensed with 4:4′-diaminostilbene-2:2′-disulphonic acid and the resulting 4:4′-bis-[4:6-dichloro-1:3:5-triazinyl-(2)-amino]-stilbene-2:2′-disulphonic acid is further condensed with aniline and *N*-methylethanolamine to yield 4:4′-bis-[4-phenylamino-6-(β-hydroxyethylmethylamino)-1:3:5-triazinyl-(2)amino]-stilbene-2:2′-disulphonic acid, which is an excellent fluorescent brightening agent for application to cellulose. C.O.C.

**Water-soluble Blue Anthraquinone Dyes for Cellulose**
ICI *BP* 772,030

Water-soluble blue dyes for cellulosic fibres



(A = subst. or unsubst. phenylene radical; R = subst. or unsubst. hydrocarbon; Y = H or $SO_3H$) are prepared by reacting an aminoanthraquinone derivative with 1 mol. cyanuric chloride. Thus, cyanuric chloride in acetone is added to ice/water and an aq. solution of the tri-Na salt of 1-amino-4-(4′-methylanilino)-anthraquinone-2:3′:5-trisulphonic acid is added to the resulting suspension at 0–2° c. The mixture is stirred and neutralised with 2N-$Na_2CO_3$. After adding $NaH_2PO_4$ and $KH_2PO_4$ the mixture is salted with NaCl. Filtration, washing with a solution containing NaCl, K and Na dihydrogen phosphate and drying gives a water-soluble blue dye which is applied to cotton or viscose in solution, the dye being finally fixed by applying an acid binding agent. E.T.

**Anthraquinone Vat Dyes**
FBy *BP* 772,042

Red-violet to blue-violet vat dyes giving dyeings of improved clarity and colour intensity are obtained when 1:5-diamino-4:8-dihydroxyanthraquinone is acylated with mixtures of *o*- and *m*- or *m*- and *p*- or *o*- and *p*-

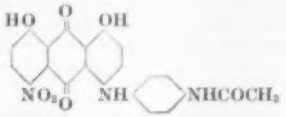
chlorobenzoic acids instead of the individual chlorobenzoic acids. A mixture of equal parts of the *m*- and *p*-isomers gives specially good results. The chlorobenzoic acid chlorides may also be used. Products are fast to light, ironing and water. E.T.

**Anthraquinone Dyes for Cotton and Synthetic Fibres**
FBy *BP* 773,212

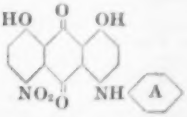
Modification of *BP* 723,079 (J.S.D.C., **71**, 196 (1955)). Dyes are produced by partially hydrolysing 1:4-diaminoanthraquinone-2:3-dinitriles in $H_2SO_4$ at 20–35°C. and reacting the products with aromatic mono- or di-amines, e.g., aniline, naphthylamine, aminoanthraquinone, naphthylene diamine. The products have higher alkali and chlorine fastness than those of the earlier process. E.T.

**Anthraquinone Disperse Dye Mixtures**
CFM *BP* 774,963

Mixtures of widely varying composition comprising—

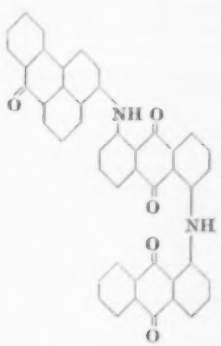


and



(A = benzene nucleus substituted by Alk or alkoxy) have much greater affinity for cellulose ester and polyamide fibres than the individual components. The dyes may be prepared separately and subsequently mixed or the mixture may be produced by mixed condensation, e.g., 1:8-dihydroxy-4:5-dinitroanthraquinone (3), *p*-aminoacetanilide (3), *p*-toluidine (2), and quinoline (20) are heated 3 hr. at 150–5°C. Mass is diluted with 15% HCl, filtered, washed with 15% HCl, and then with hot water. The product dyes cellulose acetate rayon a bright blue fast to gas fumes. E.T.

**"Benzanthronimide Green" Vat Dyes**
FBy *BP* 773,116



or the related Bz-1-benzanthronyl-5-amino-1:1′-dianthrimide carbazole on heating with complex compounds of $AlCl_3$ with bases (e.g., pyridine, quinoline, dimethylaniline, etc.) give olive-green vat dyes of good fastness properties. Thus, Bz-1-benzanthronyl-5-amino-1:1′-dianthrimide (15 pts.) is added at 110°C. to pyridine (165) and $AlCl_3$ (75) and heated 3 hr. at 120°C. The melt is added to hot NaOH and the pyridine removed by steam. The product is freed from Al salts by boiling HCl or NaOH and finally washed neutral. E.T.

**Indolium Styryl Dyes**
DuP *USP* 2,734,901

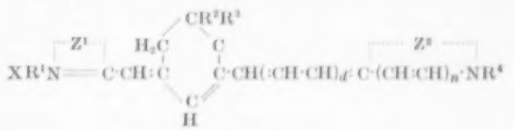
Dyes of great purity are obtained by condensing 2-methylene-1:3:3-trimethylindoline with a *p*-*N*-dialkylaminobenzaldehyde in a water-soluble alcohol, ketone, or ether, containing 5–40% by wt. of water, in presence of at least one mol. of phosphoric acid for each mol. of 2-methylene-1:3:3-trimethylindoline and separating the product from the more soluble impurities by simple filtration. Thus dimethylaminobenzaldehyde was stirred into aqueous *iso*propanol containing $H_3PO_4$ and the mixture brought to 70°C. 2-Methylene-1:3:3-trimethylindoline was then added and the mixture refluxed for 4 hr. The mixture was then cooled to 2°C., filtered, the cake washed with *iso*propanol containing $H_3PO_4$ at 4°C. and finally with *iso*propanol. The product dyed polyacrylonitrile fibres a bright deep scarlet from an aqueous bath. On heating the dyed fibres to 150–175°C. the colour only shifted slightly from scarlet towards red. C.O.C.

**Fluorescent Brightening Agents for use in White Fluorescent Marking Inks**
National Marking Machine Co. *USP* 2,734,871

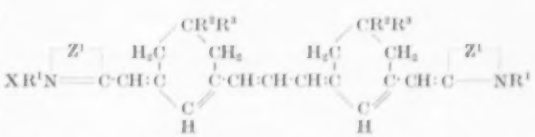
Aminostilbene derivatives having a linear aromatic polyacyl extended molecule, e.g., 4:4′-di-*p*-(benzoylaminobenzoylaminobenzoylamine) - stilbene - 2 : 2′ - disodium sulphonate and 4 : 4′ - di - *p* - (β - naphthoylaminobenzoylaminobenzoylaminobenzoyl) - stilbene - 2 : 2′ - disodium sulphonate, fluoresce white in ultraviolet radiation. They are used in laundry marking inks. C.O.C.

**Polymethin Dyes**
Eastman Kodak Co. *USP* 2,734,900

Dyes of formula—



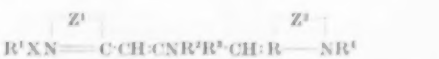
or



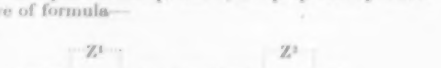
($R^1$ and $R^4$ = Alk of 1–4 C; $R^2$ = H or $CH_3$; $R^3$ = H, Alk of 1–4 C or mononuclear aromatic group of the benzene series; X = acid radical; $d$ = 1–3; $Z^1$ = atoms to complete a heterocyclic nucleus of the thiazole, benzothiazole, naphthothiazole, benzoxazole, naphthoxazole, benzoselenazole, naphthoselenazole, or thiazoline series; $Z^2$ = atoms to complete a 5- or 6-membered ring; $n$ = 0 when $Z^2$ = atoms to complete a 5-membered ring and 1 when $Z^2$ = atoms to complete a 6-membered ring), e.g., 3:3′-diethyl-9-(1-naphthyl)-thiodicarbocyanine iodide, may be used as photographic sensitisers. 25 syntheses of such dyes are given. C.O.C.

**Carbocyanine Dyes**
Eastman Kodak Co. *USP* 2,735,770

Dyes of formula—



($R^1$ and $R^4$ = Alk, $R^2$ = acyl, $R^3$ = H, Alk or an aromatic group; X = acid radical; $Z^1$ and $Z^2$ = atoms to complete heterocyclic nuclei), e.g., 9-anilino-3:3′-dimethylthiocarbocyanine *p*-toluenesulphonate, are prepared by treating a dye of formula—



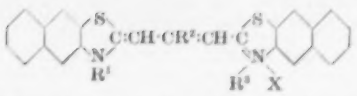
($R^5$ = Alk or aromatic) with ammonia or a primary amine of formula $R^2$-$NH_2$. C.O.C.

***meso*Phenylcarbocyanine Dyes**
Eastman Kodak Co. *USP* 2,735,767

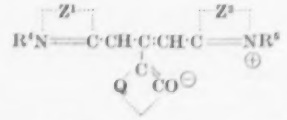
*meso*Phenylcarbocyanine dyes containing an OH group *para* to the phenyl group, e.g., 3:3′-diethyl-9-*p*-hydroxyphenylcarbocyanine iodide, are excellent photographic sensitisers. C.O.C.

**Supersensitising of Silver Halide Emulsions**
Kodak *BP* 772,906

A mixture of a dye of formula—

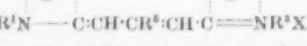


($R^1$ and $R^3$ = subst. or unsubst. Alk; $R^2$ = H or Alk; X = acid radical) and one of formula—



($R^4$ and $R^5$ = Alk; Q = atoms to complete an indanedione or a 5- or 6-membered heterocyclic nucleus; $Z^1$ = atoms to complete a benzothiazole, naphthothiazole, benzoselenazole, naphthoselenazole or naphthoxazole nucleus; $Z^2$ = atoms to complete a naphthothiazole or naphthoselenazole nucleus), e.g., a mixture of 3:3′-diethyl-5:6:5′:6′-dibenzothiocarbocyanine *p*-toluenesulphonate and 5-[(1-ethyl-2-*β*-naphthothiazolylidene)(1-methyl-2-*β*-naphthothiazolylidene)*iso*propylidene]-1:3-di(*β*-methoxyethyl) barbituric acid, is an excellent photographic supersensitiser.
*BP* 772,907

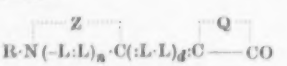
The first component may be replaced by a dye of formula—



($R^5$ = alkoxy; $Z^3$ and $Z^4$ = atoms to complete a 5-membered ring), e.g. 3:3′-dimethyl-9-methoxy-4:5:4′:5′-dibenzothiocarbocyanine bromide. C.O.C.

**Non-migrating Dyes for Photographic Emulsions**
Eastman Kodak Co. *USP* 2,735,766

Dyes of formula—



(R = Alk; L = methin; $d$ = 0–2; $n$ = 0 or 1; Z and Q = atoms to complete a 5- or 6-membered ring), e.g. 5-[3-ethyl-2(3)-benzothiazoylidene)ethylidene] - 3 - ethylrhodanine, do not tend to migrate when used in photographic emulsions. C.O.C.

**Carbon Black**
Columbian Carbon Co. *USP* 2,735,753

Improved apparatus of the type described in *USP* 2,378,055, enabling furnace black of finer particle size to be produced. C.O.C.

**Carbon Black**
Phillips Petroleum Co. *USP* 2,735,828

An improved method for recovery of furnace blacks from the furnace effluent. C.O.C.

**Stable Finely Divided Alkyl Amine Dyes**
Allied Chemical & Dye Corpn. *BP* 772,800

Modification of *BP* 760,116 (J.S.D.C., **73**, 70 (Feb. 1957) ). Addition of small amounts of the activated silica gels to salts of dye acids with alkylamines of formula $NR^1R^2R^3$ ($R^1$ and $R^3$ = Alk of 4–20 C; $R^2$ = H or Alk of 4–20 C), yields pigments which do not tend to cake on storage. Thus the salt formed by C.I. Acid Yellow 11 with di-(2-ethylhexyl)amine normally becomes a solid mass if stored for 1–3 months whereas if the pigment before being dried is mixed with activated silica gel and then dried and ground the product is still a free flowing powder after 8 months' storage. C.O.C.

**Non-dusting Powders**
ICI *BP* 772,801

The tendency to dust of finely divided water-soluble or water-dispersible powders is reduced by mixing them with an aralkyl alcohol which has solubility in boiling water of < 0·2%, e.g. *α*- or *β*-phenylethanol or *α*-phenyl-*iso*propanol. C.O.C.

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Improved Stability of Diazo Compounds (IX p. 409)
Heterocyclic *o*-Quinone Diazides for use in Diazotype Printing (IX p. 410)

# V— PAINTS; ENAMELS; INKS

PATENTS

**Introducing Metal Powders into Solvents and Dispersion Media**
J. Eckart and C. Eckart *BP* 772,841

The metal powder is encased in a material which dissolves in the solvent or dispersion medium used, the package as a whole being added to the solvent, etc. when required for use. C.O.C.

**Printing Inks**
Sun Chemical Corpn. *USP* 2,736,719

Inks of excellent workability on offset printing presses, which form extremely hard and durable films on metal, and whose hue is unaffected on baking at 360°F. contain an epichlorohydrin-bisphenol resin as the binder and a liquid chlorinated biphenyl as the carrier. C.O.C.

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Fluorescent Brightening Agents for use in White Fluorescent Marking Inks (IV p. 405)

# VI— FIBRES; YARNS; FABRICS

**Preliminary Report on Service Uses of Cyanoethylated Cotton Products**
J. Compton

*Text. Research J.*, **27**, 222–231 (March 1957)

Data so far obtained from a market evaluation programme for cyanoethylated cotton products are presented on the basis of the improved properties possessed by the fibre. Economic aspects of the large-scale production of cyanoethylated cotton are discussed. J.C.F.

**Preliminary Report on Fully Acetylated Cotton**
E. M. Buras, S. R. Hobart, C. Hamalainen, and A. S. Cooper

*Text. Research J.*, **27**, 214–222 (March 1957)

Cotton can be fully acetylated, whilst still retaining its fibrous form, by treatment with acetic anhydride in amyl acetate with perchloric acid as catalyst. The product possesses good tenacity and abrasion resistance. It has some thermoplasticity, but can be ironed below 250°c. The fibre has low regain and can be dyed with ordinary disperse dyes. Preliminary work indicates that the acetylation process can be carried out in available commercial stainless steel equipment. J.C.F.

**Crystallite Orientation in Different Varieties of Jute Fibres of Known Strength**
R. K. Sen and S. K. Chowdhury

*Text. Research J.*, **27**, 193–195 (March 1957)

The orientation of micelles in jute fibres of different varieties, having different values of intrinsic strength and quality ratio, has been studied. The Hermans' orientation factor, average angle of orientation, and angle at 40% intensity were determined for ten different samples. Orientation factor and angle at 40% intensity have no overall correlation with intrinsic strength, but there is slight correlation if the two varieties, White and Tossa, are taken separately. For the same strength, these two varieties have different orientation factors. The quality ratio of the yarn has no correlation with the orientation factor and the angle at 40% intensity. In the case of jute fibres, the average angle of orientation varies from 8 to 9° and the Hermans orientation factor from 0·96 to 0·97. For ramie, the corresponding values are 7°36′ and 0·973. J.C.F.

**Ortho–Para Cortical Differentiation in "Anomalous" Merino Wool**
N. Ahmad and W. R. Lang

*Australian J. Biol. Sci.*, **10**, 118–124 (Feb. 1957)

"Anomalous" or "doggy" merino wool appears to be an increasing problem in Australian wool growing. The pattern of the ortho–para cortical differentiation of the straighter-fibred anomalous growth has been compared with that of normal wool. It was found that demarcations of ortho- and para-cortex typical of much coarser fibres may be present in the fine fibres of anomalous growth. In general, doggy wool is coarser than the normal flock mean and has a higher % occurrence of paracortex in the cross-section. P.G.M.

**Increased Thioglycollate Extraction of Wool Proteins following Supercontraction**
J. M. Gillespie and F. G. Lennox
*Australian J. Biol. Sci.*, **10**, 95–104 (Feb. 1957)

Supercontraction of wool or of wool cells by heating in 9M-LiCl or 8M. phenol facilitates the subsequent extraction of the proteins with alkaline 0·1M. thioglycollate solutions during 20 min. at 50°C. The amount of protein extracted with 0·1M. thioglycollate initially at pH 10·0 for example, approximates to that removed from untreated wool with 0·1M. thioglycollate initially at pH 10·5. The electrophoresis patterns of the proteins extracted from supercontracted wool also resemble the patterns of extracts of normal wool obtained at higher pH values. This, together with evidence for the absence of amide bond splitting, suggests that fractional extraction with alkaline thioglycollate yields proteins pre-existing in the wool and not formed during extraction. The electrophoresis patterns would therefore appear to reflect the protein composition of reduced wool. Extraction of supercontracted wool with alkaline thioglycollate allows the wool to revert rapidly to the original uncontracted state having low thioglycollate extractability. Partial reversal of extractability is likewise achieved by prolonging the period of heating in the LiCl solution and complete reversal by heating the LiCl-supercontracted wool in water.
P.G.M.

**Fractionation of *S*-Carboxymethylkerateine 2 from Wool**
J. M. Gillespie
*Australian J. Biol. Sci.*, **10**, 105–116 (Feb. 1957)

The solubility relations of the *S*-carboxymethyl derivative of kerateine 2 (a soluble protein extracted from reduced wool) are studied in systems containing salts in high concentration, mixtures of ethanol and water, and low concentrations of certain divalent cations. Further purification of this major wool protein is obtained by fractional precipitation with zinc acetate or ammonium sulphate.
P.G.M.

**Molecular Weights of *S*-Carboxymethylkerateine 2 Fractions**
B. S. Harrap
*Australian J. Biol. Sci.*, **10**, 116–117 (Feb. 1957)

The molecular weights of the fractions of *S*-carboxymethylkerateine 2 have been determined by the surface balance technique.
P.G.M.

**Chemical Damage in Wool. II— Effects of Alkaline Solutions**
W. H. Houff, C. J. Wills, and R. H. Beaumont
*Text. Research J.*, **27**, 196–199 (March 1957)

Wool fibres were immersed in buffer solutions for 24 hr. at controlled temp., and the effect on stress–strain properties in the wet state and on lanthionine formation investigated. At 25°C., no effect on the physical properties and no lanthionine formation was observed up to pH 10. Over the range of pH values 11·0, 11·5, and 12·25 the force required to produce 25% extension in the wet state decreased progressively, and the amount of lanthionine formed, as estimated by paper chromatography, was approximately proportional to this decrease. At 50°C., immersion for 24 hr. in a solution of pH 9 significantly affects the physical properties in the wet state. At 100°C. for 24 hr., loss in strength at pH 4·5 is almost as great as at pH 6·7. Paper chromatograms of hydrolysates of the samples treated at 100°C. indicated a loss of 25–40% of cystine with approx. 75% theoretical conversion to lanthionine. For wool heated in air at 150°C. for 24 hr., approx. 30% of the cystine was converted to lanthionine; no cysteic acid could be detected.
J.C.F.

**Practical Aspects of the Regeneration of Protein Fibres**
F. Happey
*Proc. International Wool Textile Research Conf. Australia*, **E**, E 431–E 438, E 601–E 602 (1955)

A survey of materials and methods for the production of protein fibres has been made. Details are given of apparatus for spinning fibres on a small scale, by a larger-batch process, and by a continuous process.
P.G.M.

PATENTS

**Inhibition of Dark Spots on Freshly Spun Viscose Rayon by Washing with a Sequestering Agent**
American Enka Corpn. *USP* 2,735,742

The packages of freshly spun yarn are washed with an aq. soln. of a compound of formula—

$$\left[\begin{matrix} -OOC(CH_2)_b \\ \\ -OOC(CH_2)_b \end{matrix}\right.\!\!>N\cdot(CH_2)_a\cdot N<\!\!\left.\begin{matrix} (CH_2)_bCOO- \\ \\ (CH_2)_bCOO- \end{matrix}\right]^{-4}, \; M^{+4-n}H^{+n}$$

(M = alkali metal or $NH_4$; $n = 0$–2; $a = >1$; $b = >0$), e.g. tetrasodiumethylenediaminetetraacetate. This prevents formation of dark spots throughout the package and reduces the overall greyness of the yarn.
C.O.C.

**Imparting Softness and Antistatic Properties to Acrylonitrile Fibres**
Chemstrand Corp. *USP* 2,735,790

An aqueous emulsion, the solid content of which is 20–50% by wt. of a non-ionic surface-active ester, 15–35% of a non-ionic surface-active polyether, 10–40% of a cationic surface-active quaternary ammonium compound, and 5–25% of a substituted carbonyl derivative is used, e.g. a 2% aqueous dispersion whose solids consist of 48% sorbitanmonopalmitate, 32% condensate of castor oil with 200 mol. ethylene oxide, 10% *N*-cetyl, *N*-ethylmorpholiniumethosulphate and 10% $\alpha\beta$-dihydroxyethyl-$\alpha'\beta'$-di($\beta$-stearamidoethyl)urea acetate is used as an antistatic spinning lubricant.
C.O.C.

**Coating and Dyeing Textile Fibres**
T. C. Whitner *USP* 2,733,975

Modification of *USP* 2,417,388, 2,565,832 (*J.S.D.C.*, **68**, 196 (1952)), 2,646,372 (*J.S.D.C.*, **70**, 426 (1954)), and 2,675,326 (*J.S.D.C.*, **71**, 354 (1955)). The fibres are impregnated with an aqueous solution of a copper complex and sericine and then washed with an aqueous alcoholic solution of an acidic agent, e.g. HCl, and finally with water. This results in fibres which have gained more in weight, are smoother and fuller in handle, and dye more deeply than fibres obtained by the previous processes which used an aqueous solution of an acidic agent.
C.O.C.

**Polyamide Hosiery Effect Yarns**
N. V. Onderzoekingsinstituut Research *BP* 774,966

The heels and toes of hosiery are knitted with effect yarns produced by including 0·05% by weight carbon black along with the titanium dioxide during yarn manufacture.
G.E.K.

Reactions of Diazonium Compounds with Amino Acids and Proteins (XII p. 412)

## VII— DESIZING; SCOURING; CARBONISING; BLEACHING

**Soiling Characteristics of Textile Fibres. II— Influence of Fibre Geometry on Soil Retention**
A. S. Weatherburn and C. H. Bayley
*Text. Research J.*, **27**, 199–208 (March 1957)

Samples of man-made fibres were tumbled in the dry state with screened vacuum cleaner sweepings, and, after removing loose soil, were dissolved in a suitable solvent. The optical density of the suspension of soil particles was measured. Soil retention increases with decreasing filament denier, and for a given type of fibre of given denier, fibres with circular cross-section retain less soil than those with serrated cross-section. For a particular type of fibre, soil retention is a linear function of total surface area. The influence of static electrification on soiling is complex: with Terylene a pronounced tendency for electrification is associated with a high degree of soil retention, whereas with nylon an appreciable tendency for static effects is not accompanied by the expected degree of soil retention.
J.C.F.

**Physical Chemistry of Textile Scouring**
W. Kling
*S.V.F. Fachorgan*, **12**, 62–79 (Feb. 1957)

The following points are discussed— the nature of the soiling medium, the textile material which is soiled, the

type of water and scouring agent employed, the influence of surface activity in scouring, wetting out of the material and penetration, and subsequent removal of solid soil from the fabric surface. B.K.

**Modern Additions to Dry-cleaning Solvents and their Action**
H. Ewald

*S.V.F. Fachorgan*, **12**, 101–105 (Feb. 1957)

Additions to dry-cleaning solvents should pick up dirt readily, function completely within 10 min., promote the porosity of the filter cake, be readily compatible with the solvent, be transparent and cheap, have no deleterious effect on subsequent impregnation and combine with added moisture. The latter requirement arises in dry cleaning after water has been added to the dry-cleaning solvent in order to remove water-soluble impurities from the goods to be dry-cleaned. Control of the water content is described. B.K.

**Scouring of Raw Wool**
F. O. Howitt

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 315–E 330, E 587–E 588 (1955)

Preliminary experimental and technical investigations of the scouring of raw wool (mainly combing wools) are described. Consideration is given to some physico-chemical aspects of the detergent action of soap-soda systems, e.g. surface action, buffering power and effect of pH, especially in relation to commercial scouring. Mechanical factors in scouring and analytical methods related to scouring are also considered. P.G.M.

**Solvent Scouring of Raw Wool**
J. Lindberg and S. Ekegren

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 342–E 346, E 588–E 592 (1955)

A plant for the scouring of raw wool with petroleum fractions has been constructed in Sweden. The plant consists of one unit for scouring, one centrifuge for the scoured wool, three units for sludge separation and one stripping column for solvent recovery and grease separation. The economy of this scouring plant is judged to be better than that of a corresponding ordinary scouring plant. Up to 95% of the lanolin is recovered from the stripping unit. The sludge from the last separation stage is almost free from solvent and grease and can go into the sewer without danger of stream pollution. The solvent-scoured wool gives 20% longer fibres after carding and less waste. P.G.M.

**Solvent Degreasing of Wool**
J. F. Sinclair

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 347–E 362, E 588–E 592 (1955)

Laboratory experiments showed that pressure jetting of raw wool with white spirit rapidly reduced its wax, suint, and dirt content. By comparison, wool immersed and gently agitated in white spirit retained a high % of suint and dirt. A pilot plant for jet extraction of wool was constructed and its main features are described. The plant continuously degreases raw wool by extraction with cold white spirit followed by cold water extraction. Ten tons of wool have been treated with consistently less felting than in soap and soda scouring. A high % of wax is recoverable, and dirt from the wool is separated as a disposable cake; pretreatment of effluent is eliminated. The floor area occupied by the plant is substantially less than that of an emulsion scour of equivalent capacity. Capital and production costs are competitive. P.G.M.

**Action of Hexamethylenetetramine on Greasy Wool and its Effect on Scouring and Carbonising**
M. Oku and I. Shimizu

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 384–E 388 (1955)

Greasy wool prior to scouring and carbonising has been formalised using an acidic solution of hexamethylenetetramine (HMTA) in the presence of a non-ionic surface-active agent. The reaction takes place at temperatures as low as 35°c. Greasy wool is appreciably modified under these conditions, becoming alkali resistant and, at the same time, almost degreased. Damage during carbonising is considerably reduced if the wool is first formalised with HMTA. P.G.M.

**Carbonising Investigations**
**II—Uptake of Sulphuric Acid by Wool and by Trefoil Burr**
T. A. Pressley

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 389–E 397, E 596–E 599 (1955)

A number of samples of acidified wool from commercial carbonisers have been examined; the average squeeze rollers leave 67% of liquid on the neutral, oven-dry wt. of wool (40% on the wet wt.). Immersion of carded wool in acid of various concentrations, followed by centrifuging to this liquid retention, gave an uptake curve in agreement with published results, but appreciably above that of commercial samples which average 8·0% $H_2SO_4$ on the neutral, oven-dry wt. after removal from a liquid containing 5·7% w/v $H_2SO_4$. Small differences in acid concentration or squeeze roller efficiency have a minor effect on acid uptake by wool as a large proportion of the acid is held by the wool acting as base. The uptake of acid by the common trefoil burr *Medicago minima* has been followed while varying the source of the burr, pretreatment, acid concentration, and immersion time. Of these factors the source of the burr has a much greater influence on acid uptake than scouring the burr or increasing the time of immersion from 2–30 min. Burrs of identical species from different localities absorb amounts of $H_2SO_4$ varying between 2·8 and 10·2% under identical conditions. Wetting agents in the acid liquor do not affect acid absorption but alter the amount of excess liquor removed under laboratory conditions by a centrifuge or a domestic wringer. P.G.M.

**III—Protecting Wool during Carbonising**
W. G. Crewther

*Ibid.*, **E**, E 408–E 420, E 596–E 599

Wool is protected during laboratory carbonising experiments by the addition of wetting agents to the acid liquor. Anionic, cationic, and non-ionic agents are equally effective and provide adequate protection over a wide range of concentrations. Preliminary experiments to determine the nature of the reaction of sulphuric acid with wool, and of the protective action of wetting agents, are reported. P.G.M.

**Absorption of Sulphuric Acid by Burrs and its Significance in the Carbonising Process**
G. Jones

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 398–E 407, E 596–E 599 (1955)

A comparison has been made of the rate of absorption of $H_2SO_4$ by wet and dry seed pods of *Medicago minima*, the common contaminant of Australian wool, and of the equilibria attained. It was found that the rate of acid sorption by naked burrs is slower if they are dry than if wet, and the equilibrium level is a little lower, but the difference is not important after about 8 min. Acid contents after immersion under varying conditions have been recorded, both of groups of burrs as well as of individuals. The effect of various wetting agents on the acid sorption of naked burrs has been studied and found to effect little, if any, improvement, some probably exerting a retarding influence. Increase in temperature increases sorption by dry burrs. Burrs in shorn wool should be all carbonisable provided they have received at least 6–8 min. soaking after making contact with the acid. Water extraction at 80°c. of burrs from shorn wool increases the rate of sorption by dry burrs. Burrs picked from lime-painted slipe wool, even though no encrustation was discernible to the eye, sometimes showed a tendency to absorb $H_2SO_4$ slowly. P.G.M.

**Peroxide Bleaching of Cotton in the Kier**
I. S. Petrov, N. V. Sobolev, M. R. Tsimmel'zon, and V. A. Pavlova

*Tekstil. prom.*, **17**, 40–41 (March 1957)

Peroxide bleaching of coarse staple fabrics is carried out in the kier at 90°c. by the one-bath method in presence of NaOH and $Na_2SiO_3$. It is in many respects superior to hypochlorite bleaching but leads to reduced tensile strength compared with the latter method. G.J.K.

PATENT

**Dry Cleaning**
M. Blau Manufacturing Co. *USP* 2,736,632

A process in which the goods are steamed under pressure and then sprayed, also under pressure, with solvent

containing a dry-cleaning soap, rinsed and dried. This secures quickly, efficiently, and economically the advantages of both wet and dry cleaning without their limitations. C.O.C.

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Recovery of Lanolin from Wool Scour Liquors (III p. 398)

## VIII—DYEING

### Theory of Dyeing
A. Yabe

*Yûki Gôsei Kagaku Kyôkaishi*, **14**, 190–199 (1956)

Review, 55 references. C.O.C.

### Theories of Fastness Properties of Dyes
T. Iijima

*Yûki Gôsei Kagaku Kyôkaishi*, **14**, 199–204 (1956)

Review, 44 references. C.O.C.

### Engineering Principles in Dyeing and Wet Finishing
J. Lindberg

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 11–E 16, E 564 (1955)

Some of the engineering principles involved in dyeing and wet finishing are discussed. P.G.M.

### Changes in the Properties of Wool in Dyeing and Related Processes
R. V. Peryman

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 17–E 33, E 565 (1955)

The conditions used industrially for treating wool in solutions at the boil are discussed, and the factors pertinent to simulation of those processes on a laboratory scale are examined with a view to the measurement of changes produced in wool. Measurements of the pH value of the liquors after cooling are shown to be inadequate for indicating the pH values of many liquors at 100°c., and data are given for corrections which may be applied to pH values of some solutions at 18°c. Preferred methods for the physical and chemical examination of the treated wool are given. The effect on 64s-quality yarn of boiling for 3 hr. at controlled pH values in solutions containing (i) sodium sulphate and sulphuric acid or sodium carbonate, or (ii) ammonium salts and ammonia, are interpreted in terms of pH values at 100°c., and inferences having a bearing on dyehouse practice are drawn. P.G.M.

### Chroming of Wool
R. V. Peryman

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 34–E 41, E 565 (1955)

Wool yarn (64s-quality) was boiled for 3 hr. in a laboratory dyeing machine with $K_2Cr_2O_7$ (3% on the wt. of the wool) at selected constant pH values in the range 2·3–8·9. A parallel series of treatments contained $Na_2SO_4$ (1·5 g./litre) in addition to the dichromate. $Na_2SO_4$ was found to decrease the uptake of chromium at pH values below ca. 6·7 and increased it above this pH. A maximum in chromium uptake was found at pH ca. 5·5 which is also near the pH value at which a minimum amount of $Cr^{(III)}$ was found in the liquor. No evidence was found of direct attack by chromic acid or chromate on disulphide linkages in wool; it is suggested that reduction of hexavalent chromium at pH values greater than 4 is due to the products of disulphide breakdown, which occurs independently of hexavalent chromium being present except in so far as the latter behaves as a salt. P.G.M.

### Problems in Hat Dyeing
H. G. Fröhlich

*S.V.F. Fachorgan*, **12**, 80–101 (Feb. 1957)

After an introduction on raw materials and dyes, the usual types of apparatus for hat dyeing is described in detail and the advantages and disadvantages of the various machines are discussed. The influence of dyeing temp., duration of dyeing and machinery on the quality of the final goods are discussed based on both published work and the author's experiments. The dyeing behaviour of uncarrotted and carrotted furs are compared as the type of carrotting used and its concentration have an appreciable influence on the affinity of rabbit fur for acid wool dyes and on the fastness properties of the resultant dyeings. Reference is made to the causes of dyeing faults and ways of overcoming them. B.K.

### Theory of Dyeing Polyester and Polyacrylic Fibres
W. R. Remington and H. E. Schroeder

*Text. Research J.*, **27**, 177–185 (March 1957)

In the copper process for dyeing polyacrylonitrile fibres, cuprous ions complexed with –CN groups provide cationic sites on the fibre. Copolymerisation of acrylonitrile with 6% of vinylpyridine produces a fibre with affinity for anionic dyes; equilibrium studies indicate that two types of site are present. Orlon acrylic fibre can be dyed with basic dyes; the active sites appear to be acidic groups introduced during polymerisation which are neutralised by colourless cations in the undyed fibre. In the dyeing of polyester fibres with disperse dyes, no specific sites are involved, and the process is found to involve simple solution of dye in the fibre. The inconveniently slow rate of dyeing of polyester fibres under ordinary conditions may be increased by pressure dyeing at 120°c., or by the use of aromatic compounds as "carriers". Their effect is shown to be independent of the carrier structure but proportional to the number of moles of carrier dissolved in the fibre. J.C.F.

PATENT

### Striped Fabric
Springs Cotton Mills *USP* 2,733,974

An aqueous solution which either (*a*) imparts the desired colour and/or (*b*) acts as a resist for dye subsequently applied to the whole fabric, is applied. Bleeding between the stripes and the rest of the fabric is obviated by saturating the fabric with a dil. solution of dye immediately the striping liquid has been applied. The striping liquor may be applied by means of discs which rotate at a speed different from that of the fabric so that the fluid is wiped off their edges onto the cloth. C.O.C.

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Hosiery Setting and Dyeing (I p. 398)
Stable Ultraviolet Absorbers (III p. 398)
Coating and Dyeing Textile Fibres (VI p. 407)
Reactions of Diazonium Compounds with Amino Acids and Proteins (XII p. 412)

## IX—PRINTING

### Improved Stability of Diazo Compounds
A. M. Sporkin and K. I. Makarova

*Tekstil. prom.*, **17**, 52 (March 1957)

The use of chalk as neutraliser in the preparation of thickened solutions of diazo compounds in printing improves the stability of the preparation but leads to decreased (5–8%) yields of the diazo compounds. G.J.K.

### Development of Colour Photography in Europe since 1945
H. Berger

*Photo-Tech. u. Wirtsch.*, **8**, 101–102 (1957)

### Mutual Influence of Black-and-white and Colour Developers in Layers containing Colour Couplers
V. Veelen

*Sci. et Ind. phot.*, **28**, 83–85 (1957); *Chem. Abs.*, **51**, 6411 (10 May 1957)

Study of the rate of silver and dye formation in colour development by mixtures of *p*-aminodiethylaniline and black-and-white developing agents of different ionic charge. C.O.C.

PATENTS

### Squeegees for Screen Printing
Chambon *BP* 772,711

The squeegee has in its surface ink-carrying recesses arranged in succession in the direction in which the squeegee moves. This causes the squeegee to wipe the screen and so force the printing paste through it. C.O.C.

### Inhibiting Lake Formation in Printing Pastes
BrC *BP* 772,476

Lake formation, with consequent progressive weakening of the resulting prints as printing proceeds, in pastes containing direct dyes and a copper compound capable of converting the dye into a copper complex compound, is inhibited by including in the paste an alkali metal phosphate in which the molecular ratio of alkali metal oxide

to $P_2O_5$ is $< 3:2$. Thus when printing with a paste containing C.I. Direct Blue 77, a copper salt and a cationic resin fixing agent, addition to the paste of sodium hexametaphosphate leads to prints of the same hue and depth being obtained irrespective of the age of the paste.
C.O.C.

**Printing on Hats**
Geo. W. Bollman & Co. *USP* 2,736,633
The female portion of a hat mould is lined with a printing putty containing dyes, the putty being distributed through the lining so as to form a continuous design. The wet hat body is then placed in the mould and forced by the male mould member against the putty to effect transfer of dye from the putty to the hat, the time needed for this varying from 6 sec.–5 min. depending upon the amount of water in the hat and the temperature. The hat is then removed and steamed to fix the dye. The method is specially suited for applying acid dyes to wool or fur hats.
C.O.C.

**Giving Polyethylene Affinity for Printing Inks**
British Cellophane *BP* 772,803
Treatment of the surface of polyethylene with either a 4% aq. soln. of a permanganate containing 10–30% by wt. of $H_2SO_4$, or a 30% aq. soln. of a chromate or dichromate containing $> 30\%$ of $H_2SO_4$, or with a $< 35\%$ aq. soln. of $CrO_3$, washing and drying, renders the surface receptive to printing inks. When permanganate is used the brown stain can be removed by treatment with $KHSO_3$ without impairing the enhanced affinity for printing ink.
C.O.C.

**Rapid Drying of Prints**
Hardman & Holden *BP* 772,813
The drying of prints made with an ink containing a carboxy and/or hydroxyl group is accelerated by atomising a solution of an aluminium alkoxide or derivative thereof either on to the surface to be printed or on to the freshly printed material. Thus sheets of paper printed with a linseed stand oil printing ink are sprayed immediately after printing with an atomised 20% soln. of disluminium tetra*iso*propoxide-monodipropylene glycollate in mineral spirit. The sheets of paper can be piled up at once without any fear of marking off occurring.
C.O.C.

**Multicolour Printing of Continuous Metal Bands**
A. V. Borsch-Madsen *BP* 772,969
The bands are printed with each colour in succession, being heated after each colour is applied and then cooled to a constant predetermined temperature immediately before the next colour is applied. This ensures accurate registration of the various portions of the design.
C.O.C.

**Copying**
ICI *BP* 772,198
A copy sheet moistened with a duplicating fluid is pressed against a master sheet imprinted with a wax composition forming in reverse script the matter to be copied. Either the moistened copy sheet or the wax composition contains a metal compound and the other contains a substance which reacts with the metal compound to form a coloured or black metal co-ordination compound. Thus the wax composition may consist of a mixture of Carnauba wax, mineral oil and 8-hydroxyquinoline while the copy paper is moistened with an alcoholic solution of $FeCl_3$.
C.O.C.

**Heterocyclic *o*-Quinone Diazides for use in Diazotype Printing**
Kalle & Co. *BP* 772,517
The product formed by diazotising a 3-amino-4-hydroxy quinoline compound in which the benzene ring of the quinoline system contains at least one substituent or is present in a condensed form with other aromatic rings, e.g. 6:7-benzoquinoline, and then treating the resulting diazonium salt with an alkali, is used as the light-sensitive component in diazotype compositions, etc. They are especially suitable for use on aluminium in the production of printing plates.
C.O.C.

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Fluorescent Compounds for Distinguishing Photographic Materials (IV p. 402)
Supersensitising of Silver Halide Emulsions (IV p. 405)
Striped Fabric (VIII p. 409)

# X—SIZING AND FINISHING

**Proofing of Cotton**
A. B. Kogan
*Tekstil. prom.*, **17**, 39–40 (March 1957)
Low-boiling fractions of heavy wood tar are mixed with wax emulsions (4 : 1) and used for the rot- and waterproofing of cotton piece goods. The fractions contain $< 10\%$ water and $> 12\%$ phenol.
G.J.K.

**Protective Treatments for Cotton Awnings**
R. J. Bryson, W. N. Berard, and J. V. Bailey
*Text. Research J.*, **27**, 209–213 (March 1957)
The effect of outdoor weathering under sub-tropical, non-industrial conditions on cotton awnings subjected to both experimental and proprietary weather-resistant treatments is reported. A treatment consisting of 6% urea-formaldehyde resin and copper-8-quinolinolate was shown to give greater durability than standard commercial treatments. Fabric coatings based on alkyd resins proved superior to those based on natural drying oils. Treatment of sewing threads by vat dyeing followed by acetylation (Berard *et al.*, *Ibid.*, **26**, 81 (1956)) can increase the effective life by 100%. The pigmentation of awnings also affects their durability.
J.C.F.

**Water-resistant, Water-vapour-permeable Cotton Fabrics**
L. Fourt and F. Quinn
*Text. Research J.*, **27**, 232–243 (March 1957)
The water-vapour-permeability of cotton is used to greatest advantage in plastic-coated, water-resistant fabrics when the coating is relatively light and as far as possible located inside the fabric, with the cotton exposed on each surface. A plasticised resin latex with a substantial proportion of water-soluble thickener tends to give more porous films than those produced by plastisols, but each method can give a wide range of combinations of properties. The fabrics show vapour and hydrostatic resistances which compare well with breathable vinyl fabrics, but the appearance and feel are different.
J.C.F.

**Permanent Creasing of Cotton Garments**
J. D. Reid, L. W. Mazzeno, R. M. Reinhardt, and A. R. Markezich
*Text. Research J.*, **27**, 252–259 (March 1957)
A 4% solids uptake of a dimethylol cyclic ethyleneurea formulation compounded with an acrylic thermoplast applied to cotton fabric and garments gives permanent creases which will withstand at least twelve simulated launderings. Curing and creasing can be done with a domestic electric iron. Variables studied include the effect of storage on impregnated cloth, possibilities of stripping, precautions in drying and curing, effect of sizing, effect of catalysts, and changes in the resin formulation.
J.C.F.

**Application of Wrinkle-resistant Finishes to Cotton Garments**
M. D. Hurwitz
*Text. Research J.*, **27**, 267–270 (March 1957)
A conventional crease-proofing treatment suitable for application to cotton garments in the home is described. The finish is based on a methylol cyclic urea resin with a metal salt catalyst, and curing is done by ironing.
J.C.F.

**Application of Crease-holding, Wrinkle-resistant Treatments to Cotton Garments by Dry-cleaners**
R. T. Graham
*Text. Research J.*, **27**, 270–273 (March 1957)
A description of preliminary work on the application of a permanent crease finish to cotton garments using conventional dry-cleaning plant.
J.C.F.

**Flame-retardants for Cotton using Resins based on Tris(1-aziridinyl)phosphine oxide (APO) or Tris(1-aziridinyl)phosphine sulphide (APS) with Tetrakis-(hydroxymethyl)phosphonium chloride (THPC)**
W. A. Reeves, G. L. Drake, L. H. Chance, and J. D. Guthrie
*Text. Research J.*, **27**, 260–266 (March 1957)
Fabric is padded in an aqueous solution of APO and THPC, or APS and THPC, dried, cured at 140°C. for 5 min., and rinsed to remove unpolymerised material. The padding bath is stable, and the amount of resin solids required for a permanent finish is low in comparison with other durable flame retardants. Treated fabrics have an

unimpaired handle, and good tensile and tear strength. The finish is very resistant to neutral and alkaline washing, and the strength of treated fabrics is not impaired by chlorine bleaching. J.C.F.

**Prevention of Felting with Resins and the Significance of Pretreatment**
D. L. C. Jackson

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 439–E 451, E 603–E 607 (1955)

Various types of resins have been applied to wool which had received different pretreatments and the effects on felting were determined. Resins which were applied in the preformed or polymerised state (poly-amide, casein, melamine-formaldehyde) were not effective in eliminating felting unless the wool had received a pretreatment. These materials rely on forces of adhesion for their effect and contaminants on the fibre surface reduce these forces. Pretreatment of the wool by ethanol extraction, or with chemical agents which modify the fibre surface, increase the effectiveness of the resins. Polyglycine which was applied in such a way that it was covalently attached to the wool, was equally effective whether or not a pretreatment was used. An organo-silicon resin, which was polymerised in presence of the wool, behaved as the preformed resins in that it was not effective unless the wool had been pretreated. The resin is initially attached to the fibre via the amino groups but the bond is subsequently hydrolysed during washing, and so the resin depends on its adhesion to the fibre for its non-felting effect. Polyurea, which was polymerised also in presence of wool, was more effective when the wool was chemically treated than when it was ethanol-extracted. It is assumed that the diisocyanate is held by virtue of partial diffusion into the fibre so that the final condensation polymer does not rely entirely on adhesive forces for its attachment to the wool.

P.G.M.

**Effect of Casein Shrink-resistant Treatments on the Properties of Wool**
D. L. C. Jackson

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 452–E 459, E 603–E 607 (1955)

Wool fabrics have been treated with $H_2O_2$ solutions of various concentrations, and then with casein which was hardened with formaldehyde. The effect of these treatments, separately and together, on felting shrinkage, alkali resistance, elastic properties, and yarn tensile strength have been determined. The most notable effects are that treatment with $H_2O_2$ reduces alkali resistance, but subsequent treatment with formaldehyde restores this to a value even greater than the original, while casein treatment increases yarn tensile strength also to a value higher than the original. Thus, treatment of wool with casein and formaldehyde for the purpose of preventing felting, not only eliminates felting shrinkage but also increases its alkali resistance and tensile strength.

P.G.M.

**Mechanisms by which Resins prevent Felting**
K. R. Makinson

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 460–E 471, E 603–E 607 (1955)

Samples of worsted fabric subjected to combinations of pretreatment and resin treatment described by Jackson (*Proc. Int. Text. Res. Conf. Australia*, **E**, E 439 (1955)) have been examined microscopically, and the frictional properties of fibres from some samples have been examined qualitatively. It has been found that there are 4 different mechanisms by which felting is prevented: (i) fibre-bonding (poly-amide, casein, melamine-formaldehyde); (ii) scale-masking by a continuous, smooth film of resin (silane); (iii) scale-masking by a rough deposit of resin (poly-urea); (iv) destruction of the frictional difference along a fibre in a way which is not yet clear, but is probably a rounding off of the scale edges by a sub-microscopically thin film of resin (poly-glycine). Jackson's conclusions as to the nature of the forces involved are in general supported. P.G.M.

**Shrink-resisting Wool**
A. N. Davidson and R. Preston

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 472–E 488, E 607–E 609 (1955)

Methods for the production of shrink-resisting wool fabrics are outlined and briefly discussed. A recently introduced modification of the Perzyme method is described, in which the hydrogen peroxide pretreatment is replaced by a treatment with dilute peracetic acid; this is followed by the normal treatment with papain or by solutions containing free chlorine. P.G.M.

**Mechanism of the Chemical Anti-felting Reactions**
C. Earland

*J. Textile Inst.*, **48**, T 132 (April 1957)

The results of Davidson and Preston (*J. Textile Inst.*, **47**, P 685 (1956)) with a two-bath anti-felting treatment by peracetic acid and sodium hypochlorite are held to support the theory of Alexander, Carter, and Earland (J.S.D.C., **65**, 447 (1951)) that anti-felting treatment involves rupture of disulphide cross-linkages and, in addition, some main-chain attack on the protein.

P.T.S.

**Pilot-scale Shrink-resistant Treatments of Wool**
J. Delmenico

**I— Application of Methoxymethylnylon**

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 489–E 501, E 609–E 612 (1955)

Pilot-scale development of the shrinkproofing of wool with an ethanolic solution of methoxymethyl nylon and KOH is described. The wool must be scoured, rinsed, dried, and conditioned before treatment. Soap or anionic detergents may be used for scouring. NaOH in equal concentrations may replace KOH. The optimum treatment times are given for concentrations of alkali from 0·5–5·0% at temperatures of 20, 25, and 30°C. The method of treatment was to press the wool (20 lb.) into a small amount of ethanol (liquor : wool ratio 1 : 1) containing the resin (2%), NaOH (2%), and water (2%) for 2½ min. The goods were placed in a rotary drier, the action of the alkali stopped by treatment with $CO_2$, and the ethanol removed at about 35–40°C. The resin was hydrolysed to nylon with aqueous 2*N* HCl, and the wool then neutralised with $Na_2CO_3$ and rinsed. P.G.M.

**II— Improved Method of Treatment with Alcoholic Alkali**

*Ibid.*, **E**, E 502–E 513, E 609–E 612

A modified method of shrinkproofing wool with ethanolic alkali has been devised and used successfully in a pilot scale. The wool was pressed into the solution using a liquor : wool ratio of 1 : 1. To avoid uneven treatment in the press, the temperature of the wool should be similar to that of the solution. For an effective treatment, the concentration of KOH should be at least 4%, the temperature 25–30°C., and the time of treatment 5–10 min. Use of such a relatively small amount of solution makes it unnecessary to recover the ethanol. This method eliminated problems associated with the steeping technique, such as the accumulation of water and impurities in the alkali solution. Instead of using aqueous or ethanolic $H_2SO_4$ to neutralise the residual alkali, the wool was treated with $CO_2$ and then rinsed in water. This reduced the amount of handling required, and eliminated staining arising from impurities in the acid bath, or from deposition of suspended matter from the tap water onto the wool while in the acid condition. Wool treated by this method showed the same felting shrinkage, tensile strength, bursting strength, and acid solubility as wool treated by the usual steeping technique and neutralised with acid.

P.G.M.

**Mothproofing of Wool**

**I— The Application of Anionic Surface-active Agents**
M. Lipson

*Proc. International Wool Textile Research Conf. Australia*, **E**, E 514–E 522, E 612–E 615 (1955)

A range of anionic surface-active agents has been found to impart moth-resistance to wool when applied at pH 2 and 50°C. Effectiveness of treatment depends on the configuration of the alkyl chain which, in aliphatic compounds, should preferentially be attached direct to the $-SO_3H$ group; chain length is also important. The surface-active agents are fast to dry-cleaning, but wash-fastness is unsatisfactory, and light-fastness is low.

**II— The Effect of Dieldrin and Related Compounds**
M. Lipson and R. J. Hope

*Ibid.*, **E**, E 523–E 530, E 612–E 615

Low concentrations of Dieldrin will protect wool from moth-larval attack even after prolonged washing and dry-

cleaning. Endrin gives similar results, but Aldrin is not as satisfactory. The effect with Dieldrin remains after outdoor exposure over a period of 4 months. Scoured wool from fleece samples taken from sheep 3 months after dipping in Dieldrin emulsions was found to be mothproof. Exposure tests are in progress over longer periods. Dieldrin is also effective against larvae of Australian and furniture carpet-beetles. P.G.M.

PATENTS

**Modifying Cellulose or Cellulose Derivatives**
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**Setting the Twist in Yarn**
Commonwealth Engineering Co. *USP* 2,734,006

The twisted yarn is treated with a dextran ester of a fatty acid of 8–18°C. in either aqueous dispersion or dissolved in a non-polar solvent. C.O.C.

**Permanent Glazed or Embossed Patterned Finish**
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Permanent glazed or embossed patterns are produced on cloth with no loss in strength even in the case of light-weight fabrics, by calendering the cloth to produce a glazed or embossed finish, applying a thermosetting resin in the desired pattern, curing the resin and then treating with an aqueous liquid to remove the calendered finish from the areas free from resin. If desired before the cloth is calendered it may be coated or impregnated with a mechanically deformable cellulosic material preferably formed *in situ*. This treatment protects the fibres against possible damage caused during calendering or by the catalyst used for the resin. C.O.C.

**Shrinking Fabric with Improvement in Handle**
C. G. Hatay *USP* 2,733,498

If a tensionless wet cloth is subjected to successive localised blows at a time when the other side of the cloth than that struck is not touching a rigid surface then the cloth is thickened and its handle improved but is not rendered polished or shiny. If the cloth is dried to < 25% moisture content while being "beaten" then the finished cloth has little or no residual shrinkage. Machinery embodying this principle is described. C.O.C.

**Enhancing the Fastness to Washing of Flame-resistant Finishes**
Ciba *BP* 772,367

The flame-resistant finish produced according to *BP* 747,014 (J.S.D.C., **72**, 365 (1956)) when treated with HCHO, especially with aq. HCHO at 80–150°C., is given improved fastness to washing, e.g. it can be given one or more alkaline washes without the flame-resistance being impaired provided that after washing the alkali metal ions left in the fibres are replaced by ions of a readily volatile base, e.g. by a final rinse in aq. ammonium sulphate. C.O.C.

**Fabrics Coated with Metal**
Heberlein & Co. *BP* 772,127

Fabric is given a permanent fine embossed effect, particularly a schreiner or moiré finish, and is then treated with metal vapour. Thus dyed nylon toile is schreinered at 200°C. and then aluminium vapour is deposited on it in a high vacuum. The resulting cloth has a strikingly beautiful sheen resembling that of silver brocade. C.O.C.

Hosiery Setting and Dyeing (I p. 398)
Stable Ultraviolet Absorbers (III p. 398)
Coating and Dyeing Textile Fibres (VI p. 407)

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

PATENTS

**Cellulose Organic Ester Moulding Powders containing Phototropic Dyes**
Eastman Kodak Co. *USP* 2,735,783

Coloration of cellulose ester plastics with phototropic dyes, e.g. *p*-[*o*-chlorophenylazo]*NN*-dimethylaniline, protects the plastics from degradation on exposure to ultraviolet radiation. C.O.C.

**Stabilised Pigmented Cellulose Acetate-Butyrate Moulding Compositions containing Propyl Gallate**
Eastman Kodak Co. *USP* 2,735,782

A combination of $TiO_2$ and propyl gallate prolongs to a surprising extent the resistance to weathering of cellulose acetate-butyrate moulding compositions. C.O.C.

## XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

**Cytochemical Diazo Reaction for Nucleoprotein**
E. A. Barnard and J. F. Danielli
*Nature*, **178**, 1450–1453 (29 Dec. 1956)

Dry tissue sections, after treatment under anhydrous conditions with benzoyl chloride, which should react with all components capable of reacting with a diazonium compound, still show a residual reaction with tetrazotised dianisidine. If water is present before benzoylation the reaction does not occur. Evidence suggests that the reaction is due to a nucleoprotein component, probably a histidine residue. It is thought that this reaction indicates a special type of association between components of the nucleus. A.J.

**Reactions of Diazonium Compounds with Amino Acids and Proteins**
A. N. Howard and F. Wild
*Biochem. J.*, **65**, 651–659 (April 1957)

Pauly (1915) showed that the phenolic group of tyrosine and the iminazole group of histidine can react with diazo reagents. Reaction with other groups present in proteins has been considered and derivatives were prepared with compounds containing certain amino groups and guanidino, thiol, and indolyl groups. From the evidence of other authors, it is also concluded that imino and aliphatic hydroxyl groups react, whereas all other groups are inert. Of the aliphatic primary amino compounds investigated, only those containing the grouping $-CH_2{\cdot}NH_2$ have been found to yield triazens. Other types of amino group were deaminated, an aromatic diazoamino compound being produced as the by-product. It is inferred that for amino acids found in proteins, only the $\varepsilon$-amino group of lysine and the $\alpha$-amino group of glycine are capable of forming bis(diazo)amino links. The maximum amount of diazotised *p*-arsanilic acid which can react with four different proteins has been determined and the values of the arsenic/nitrogen quotients were obtained by analysis. A comparison of these results with the theoretical values calculated on the basis of their known amino acid content has given an indication of the extent of the reaction. It is concluded that reaction occurs between one diazonium radical and each thiol and indolyl group; between two diazonium radicals and each phenolic, iminazole, $\varepsilon$-amino group and $\alpha$-amino group of *N*-terminal glycine residues; and between three diazonium radicals and each guanidino group. It is concluded that $\alpha$-amino groups of *N*-terminal residues other than glycidine are deaminated. P.G.M.

## XIII—RUBBER; RESINS; PLASTICS

PATENT

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## XIV—ANALYSIS; TESTING; APPARATUS

Reactions of Diazonium Compounds with Amino Acids and Proteins (XII above)

## XV—MISCELLANEOUS

**Note on the Writing of Scientific Papers and Theses**
W. Hume-Rothery
*Research*, **10**, 168–176 (May 1957)

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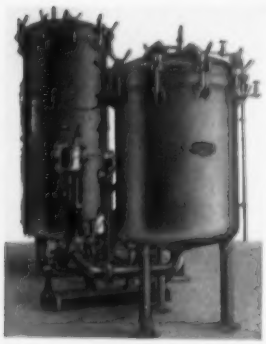

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## FORTHCOMING MEETINGS OF THE SOCIETY—*continued from page x*

**Thursday, 16th January 1958**

WEST RIDING SECTION. *Continuous Dyeing of Wool.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Victoria Hotel, **Bradford.** 7.30 p.m.

**Friday, 17th January 1958**

LONDON SECTION. Dinner and Dance. Waldorf Hotel, **London, W.C.2.**

**Tuesday, 21st January 1958**

SCOTTISH SECTION. *Flame-resistant Finishes.* J. R. W. Perfect, Esq., B.Sc. (Bradford Dyers' Association Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.
Developments with the use of phosphorus-containing resins.

**Wednesday, 22nd January 1958**

MIDLANDS SECTION. *Colloquium . . . Auxiliary products—Use and Abuse.* (Short papers by local members.) College of Technology, **Leicester.** 7 p.m.

SCOTTISH JUNIOR BRANCH. *Practical Aspects of Fastness Testing To-day.* P. R. Dawson, Esq. (Imperial Chemical Industries Ltd.). Technical College, **Paisley.** 7.30 p.m.

**Friday, 24th January 1958**

WEST RIDING SECTION. Ladies' Evening. Victoria Hotel, **Bradford.** 7.30 p.m.

**Thursday, 30th January 1958**

WEST RIDING SECTION. *A New Approach to Pigment Dyeing of Piece Goods.* Dr. Sulzer (Ciba Ltd., Basle). Victoria Hotel, **Bradford.** 7.30 p.m.

**Tuesday, 4th February 1958**

LONDON SECTION. *Progress in Textile. A review of developments and their effect on industry and user—Dyeing and Finishing.* T. Vickerstaff, Esq., M.Sc., Ph.D., F.S.D.C. (Joint Meeting with the London Section, Textile Institute.) Chemical Society, Burlington House, **London, W.1.** 6.30 p.m.

**Friday, 7th February 1958**

LONDON SECTION. *Spun-dyed Fibres, their Properties and Uses.* C. C. Wilcock, Esq., A.R.T.C.S., F.T.I., F.S.D.C. (Courtaulds Ltd.). Royal Society, Burlington House, **London, W.1.** 6 p.m.

**Tuesday, 11th February 1958**

NORTHERN IRELAND SECTION. *A New Approach to Pigment Dyeing.* Dr. G. Sulzer (Ciba Limited). Further details later.

**Thursday, 13th February 1958**

MANCHESTER JUNIOR BRANCH. *The Scientific Background to Mangling.* F. W. Thomas, Esq. Manchester College of Technology, **Manchester.** 4.30 p.m.

MIDLANDS SECTION. *A New Approach to the Pigment Dyeing of Piece Goods.* Dr. G. Sulzer (Ciba Ltd, Basle). Gas Board Theatre, **Nottingham.** 7 p.m.

WEST RIDING SECTION. *Investigations on the Dyeing Process of Polyester Fibres with Disperse Dyes.* Dr. Glenz (Bayer Co., Leverkusen). Metropole Hotel, King Street, **Leeds.** 7.30 p.m.

**Tuesday, 18th February 1958**

SCOTTISH SECTION. *A New Approach to Pigment Dyeing of Piece Goods.* Dr. G. Sulzer (Ciba Ltd., Basle). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

**Friday, 28th February 1958**

WEST RIDING SECTION. J. B. Speakman, Esq., D.Sc., F.R.I.C., F.T.I. Details later.

**Friday, 7th March 1958**

LONDON SECTION. *Recent Developments in the Dyeing of Man-made Fibres.* Dr. R. Wittwer (Ciba Ltd.). Royal Society, Burlington House, **London, W.1.** 6 p.m.

**Tuesday, 11th March 1958**

NORTHERN IRELAND SECTION. *Some Observations in the Uses of Synthetic Resin Products and Chemical Reactants to Cellulosic Materials.* F. Sloan, Esq., M.Sc. (Kirkpatrick Bros. Ltd.). Further details later.

**Thursday, 13th March 1958**

MANCHESTER JUNIOR BRANCH. *Terylene.* J. R. Whinfield, Esq. Manchester College of Technology, **Manchester.** 4.30 p.m.

WEST RIDING SECTION. Discussion, *Optical Whitening.*
Panel { A Member of the British Cotton Industries Research Association. / J. S. Ingham, Esq. (Marks & Spencer Ltd.). / Dr. Fearnley (Geigy Co. Ltd., Manchester). / J. Rayment, Esq. (G. & W. N. Hicking).
Victoria Hotel, **Bradford.** 7.30 p.m.

**Tuesday, 18th March 1958**

SCOTTISH SECTION. *Easy-care Finishes on Fabrics Containing Courpleta, with Special Reference to Pleating, Embossing and Setting.* A. S. Cluley, Esq., F.T.I. and S. N. Bradshaw, Esq. (Courtaulds Ltd., Coventry). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

**Wednesday, 19th March 1958**

MIDLANDS SECTION. *Optical Brightening Agents.* D. A. W. Adams, Esq., B.Sc., Ph.D. (Joint meeting with The British Association of Chemists.) Midland Hotel, **Derby.** 7 p.m.

**Friday, 21st March 1958**

MIDLANDS SECTION. Section Annual Dinner. George Hotel, **Nottingham.**

**Thursday, 27th March 1958**

WEST RIDING SECTION. Annual General Meeting. Victoria Hotel, **Bradford.** 7.30 p.m.

**Wednesday, 16th April 1958**

MIDLANDS SECTION. *Basic Principles of Dyeing with Disperse Dyes.* D. Finlayson, Esq., M.C., M.A., D.Sc. and H. C. Olpin, Esq., M.Sc., F.R.I.C., F.S.D.C. King's Head Hotel, **Loughborough.** 7 p.m. (Preceded at 6.30 p.m. by the Annual General Meeting of the Section.)

**Friday, 18th April 1958**

LONDON SECTION. *Colour Fastness Requirements of Luton Textiles.* I. Glasman, Esq., A.T.I. (Marks & Spencer Ltd.). George Hotel, **Luton.** 7 p.m.

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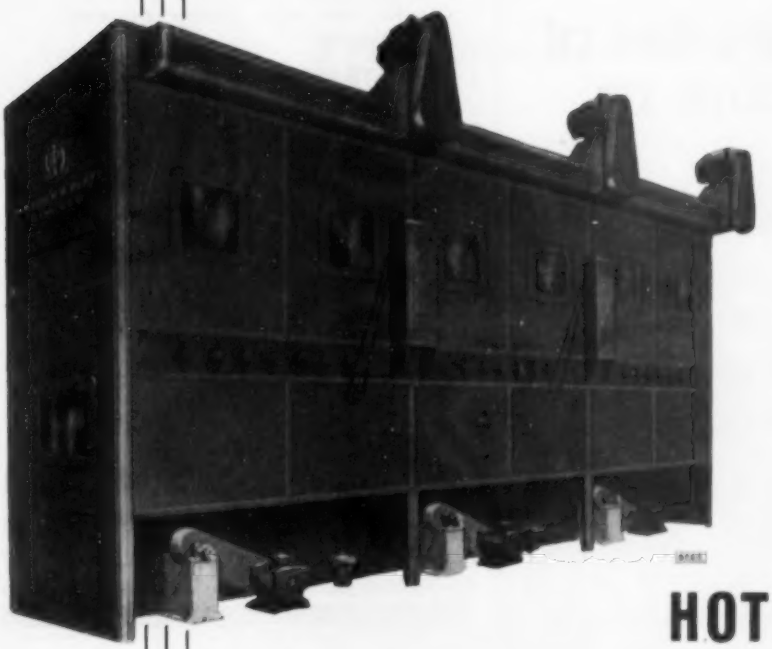

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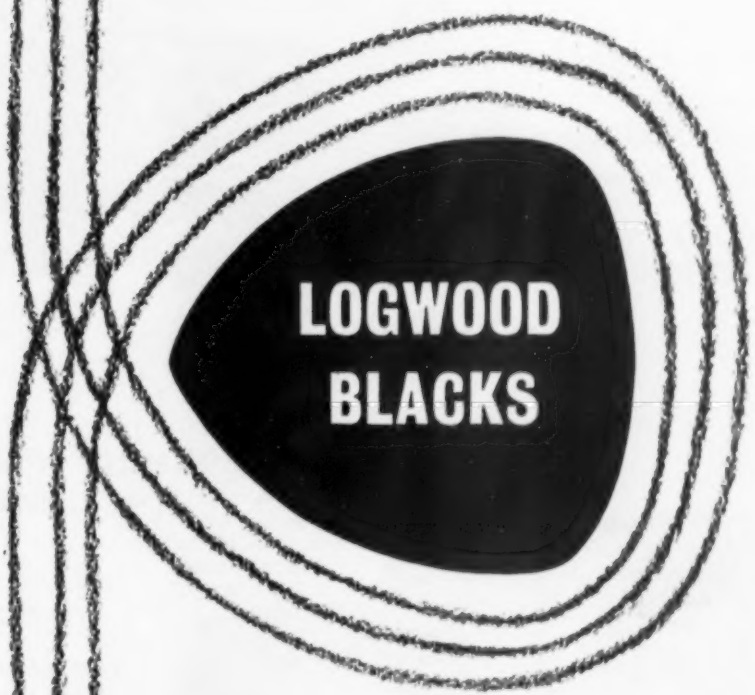


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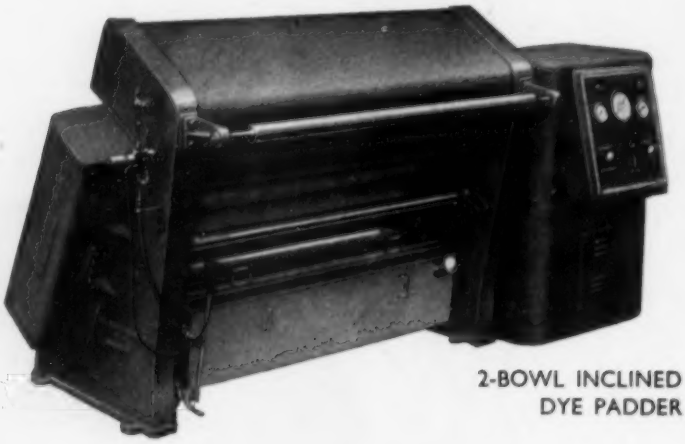

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